

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

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**BACHELOR OF CHEMICAL ENGINEERING  
UNIVERSITI MALAYSIA PAHANG**

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

**VERONEKA SEMILIN**

Thesis submitted in partial fulfilment of the requirements  
for the award of the degree of  
Bachelor of Chemical Engineering

**Faculty of Chemical & Natural Resources Engineering  
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JANUARY 2014

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I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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**Dedication**

*My special dedication for my father, Semisin Balensiu  
and my mother, Rosney Pindah,  
also to my brothers and sister*

§

*To my supervisor, Dr. Ahmad Ziad Bin Sulaiman  
for their supports and encourage.*

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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<sub>2</sub>/100g, peroxide value of 14.8 meq H<sub>2</sub>O<sub>2</sub>/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<sub>2</sub>/100g, 106.3 meq H<sub>2</sub>O<sub>2</sub>/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 mengandung protein dan vitamin yang baik untuk kesehatan 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 pengestrak 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 apparatus 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 I<sub>2</sub>/100g, nilai peroksida 14.8 meq H<sub>2</sub>O<sub>2</sub> / g, dan nilai nilai asid lemak sebanyak 49.3 % (oleik), manakala minyak bijan berkulit mengandungi 210,4 mg KOH / g, 1.34 g I<sub>2</sub>/100g, 106.3 meq H<sub>2</sub>O<sub>2</sub> / 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.

# TABLE OF CONTENTS

SUPERVISOR'S DECLARATION .....	IV
STUDENT'S DECLARATION .....	V
<i>Dedication</i> .....	VI
ACKNOWLEDGEMENT .....	VII
ABSTRACT.....	VIII
ABSTRAK.....	IX
TABLE OF CONTENTS.....	X
LIST OF FIGURES .....	XII
LIST OF TABLES .....	XIII
LIST OF ABBREVIATIONS.....	XIV
LIST OF ABBREVIATIONS.....	XV
1 INTRODUCTION .....	16
1.1 Motivation and statement of problem .....	16
1.2 Objectives.....	19
1.3 Scope of this research.....	19
1.4 Main contribution of this work .....	19
1.5 Organisation of this thesis .....	19
2 LITERATURE REVIEW .....	21
2.1 Overview .....	21
2.2 Introduction .....	21
2.3 Previous work on Sesame seed .....	21
2.3.1 Nutrient and Composition of Sesame seed .....	21
2.3.2 Fatty acids .....	23
2.3.3 Types of Extraction.....	24
2.3.4 Direct Saponification .....	25
2.4 Summary .....	26
3 MATERIALS AND METHODS.....	27
3.1 Overview .....	27
3.2 Introduction .....	27
3.3 Chemicals.....	27
3.4 Raw material .....	27
3.5 Equipments.....	28
3.6 Experimental work .....	29
3.7 Sample Preparation .....	30
3.8 Extraction of Sesame seed using Soxhlet Extractor.....	30
3.9 Physiochemical Analysis .....	31
3.9.1 Acid Value (AV).....	31
3.9.2 Saponification Value (SV).....	31
3.9.3 Iodine Value (IV).....	32
3.9.4 Free Fatty Acid Value (FAV) .....	33
3.9.5 Peroxide Value (PV).....	33
3.10 Soap making by Direct Saponification.....	34
3.10.1 Solid soap.....	35
3.10.2 Liquid soap .....	35
3.11 Soap Composition Analysis .....	35

3.11.1	The pH Value determination.....	36
3.11.2	The Foam height determination.....	36
3.11.3	The Observation of the soap.....	36
3.12	Soap Composition Testing.....	37
3.13	Summary.....	37
4	RESULT AND DISCUSSIONS.....	38
4.1	Overview.....	38
4.2	Introduction.....	38
4.3	Physiochemical Analysis of Sesame Oil.....	38
A)	Free Fatty Acid Value (FFAV).....	38
B)	Saponification Value (SV).....	40
C)	Acid Value (AV).....	41
D)	Iodine Value (IV).....	42
E)	Peroxide Value (PV).....	43
4.4	Physiochemical Analysis of Sesame Soap.....	47
4.5	Sesame oil Soap Testing.....	50
4.6	Summary.....	51
5	CONCLUSIONS.....	52
5.1	Introduction.....	52
5.2	Conclusion.....	52
5.3	Future work.....	52
	REFERENCES.....	54
	APPENDICES.....	59
	APPENDIX A.....	60
	APPENDIX B.....	74

## **LIST OF FIGURES**

Figure 1.1: The plant of sesame.....	16
Figure 2.1: Characteristic of fatty acids (Retrieved from <a href="http://www.soapcalc.net/info/SoapQualities.asp">http://www.soapcalc.net/info/SoapQualities.asp</a> ).....	24
Figure 3.3: Flow chart of Experimental Work.....	29
Figure 4.4: Sesame Seed Oil.....	46
Figure 4.5: Comparison of Sesame oil Soap.....	49

## LIST OF TABLES

Table 2-1: Proximate Composition of Sesame seed (%) .....	22
Table 2-2: Oil contents and Characteristic of Sesame seed.....	23
Table 4-3A: Data collected for Dehulled Sesame oil (FFAV) .....	39
Table 4-4A: Data collected for Undehulled Sesame oil (FFAV) .....	39
Table 4-5B: Data collected for Dehulled sesame oil (SV) .....	40
Table 4-6B: Data collected for undehulled sesame oil (SV) .....	40
Table 4-7C: Data collected for dehulled sesame oil (AV).....	41
Table 4-8C: Data collected from undehulled sesame oil (AV) .....	41
Table 4-9D: Data collected for Dehulled sesame oil (IV) .....	42
Table 4-10D: Data collected for Undehulled sesame oil (IV) .....	42
Table 4-11E: Data collected for Dehulled sesame oil (PV).....	43
Table 4-12E: Data collected for Undehulled sesame oil (PV).....	44
Table 4-13: Overall Value of Physiochemical Analysis of Sesame oil.....	45
Table 4-14: Physiochemical Analysis of Sesame soap.....	47
Table 4-15: Sesame ( <i>sesamum indicum</i> ) soap Testing .....	50

## LIST OF ABBREVIATIONS

$cm^3$	cubic centimetre
%	percentage
<i>cm</i>	centimetre
<i>mg</i>	milligram
<i>g</i>	gram
$\omega$	omega
$^{\circ}C$	temperature
<i>M</i>	molarity
<i>mL</i>	mile litre
<i>N</i>	normality
<i>meq</i>	measured in equivalent
<i>s</i>	second
<i>i.e.</i>	in example

## LIST OF ABBREVIATIONS

AV	Acid Value
CH <sub>3</sub> OH	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 <sub>3</sub>	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-

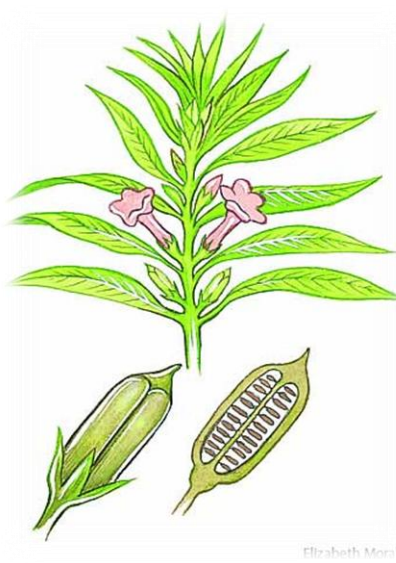


Figure 1.1: The plant of sesame



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, dehulled 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 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 include 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 (%)

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

<b>Characteristics</b>	<b>White sesame seed (wt %)</b>	
	Unroasted	Roasted
<b>Palmitic</b>	8.7	8.7
<b>Stearic</b>	4.9	5.1
<b>Oleic</b>	46.8	47.2
<b>Linoleic</b>	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-linoleic 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  $\omega$ -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).



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

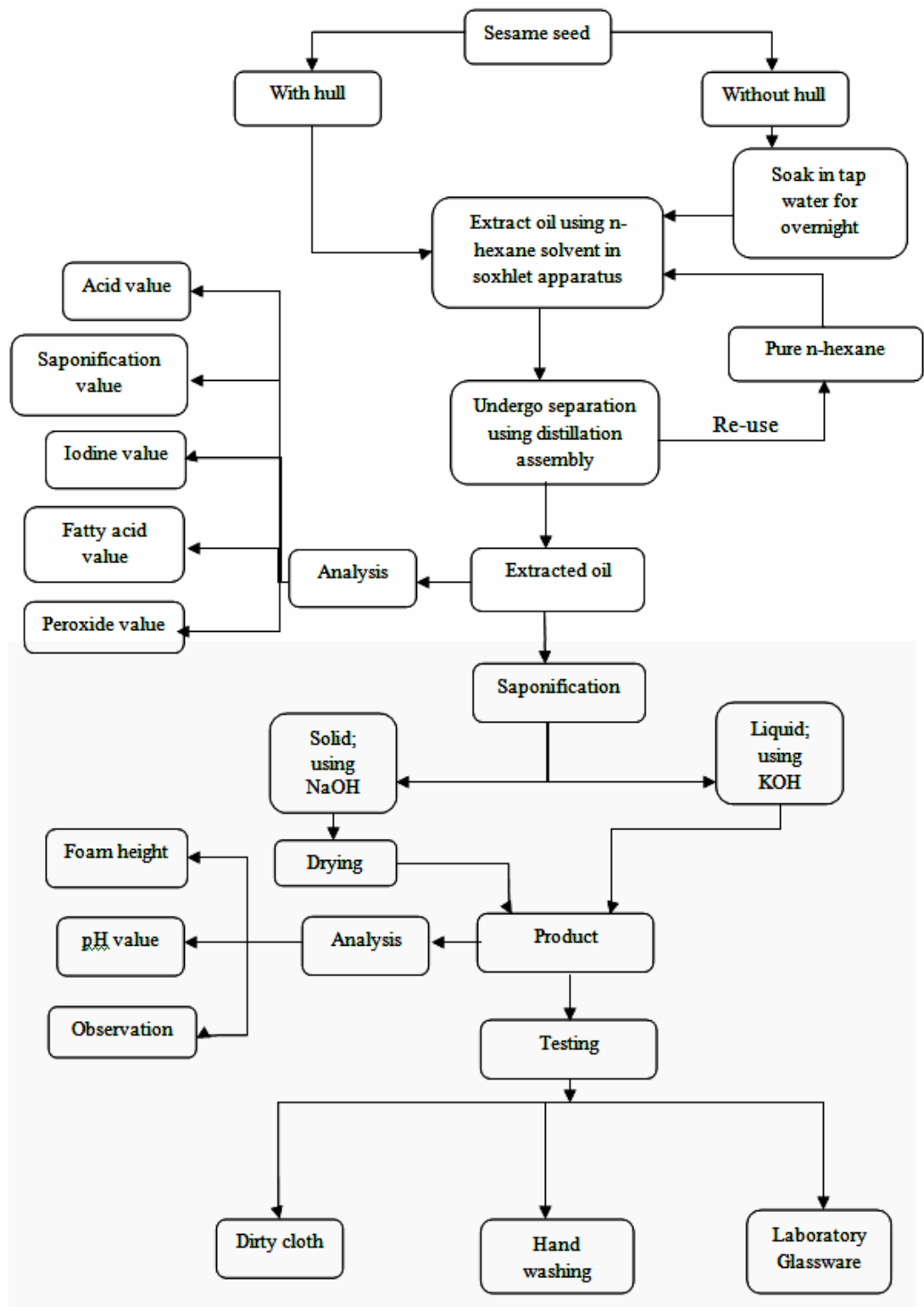


Figure 3.3: Flow chart of Experimental Work

### ***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°C for overnight. After dried, the seeds were finely grounded by using stainless steel blender. The grounded seed were dried at 45°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°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.

$$Yield (\%) = \frac{Oil\ weight}{sample\ weight} \times 100\% \quad (2)$$

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

### **3.9 Physiochemical Analysis**

The acid value (AV), iodine value (IV) and saponification value (SV) determination are carried out by using the methods reported by Warra et al (2011), Liauw (2008) and ISO 3961 standard. Free fatty acid is determined by using methods that reported by Salimon et al (2011), and peroxide value (PV) is determined by using methods that reported by Warra (2012).

#### **3.9.1 Acid Value (AV)**

50 cm<sup>3</sup> of ethanol was heated with 5 g of the oil sample in a 250 cm<sup>3</sup> conical flask to mix up the mixture. The heating was stopped and cooled, and was titrated with 0.1 M of KOH solution, by adding two drops of phenolphthalein as indicator until a permanent pink colour was obtained at the end point. Acid value was calculated using the expression;

$$A.V = \frac{5.61 \times \text{no. of mL } 0.1 \text{ M KOH}}{\text{weight, g}} \quad (3)$$

#### **3.9.2 Saponification Value (SV)**

2 g of the oil sample was added to a conical flask containing 30 cm<sup>3</sup> of ethanolic KOH and was attached to condenser for 30 minutes to ensure that the sample is fully dissolved. After sample has cooled, 1 cm<sup>3</sup> of phenolphthalein (indicator) was added and titrated with 0.5 M HCl until a pink colour indicates the end point. Saponification value was calculated using the expression;

$$S.V = \frac{(S-B) \times M \times 56.1}{\text{sample weight (g)}} \quad (4)$$

Where,

S = Sample titrate value

B = Blank titrate value

M = Molarity of the HCl

56.1 = Molecular weight of KOH

### 3.9.3 Iodine Value (IV)

0.4 g the sample was weighed into a conical flask and 20 cm<sup>3</sup> of carbon tetrachloride (purity 99%) was added to dissolve the oil. Then, 23 cm<sup>3</sup> Wij's reagent (FFSAI, 2012) was added to the flask using a safely pipette in fume chamber. Stopper was then inserted and the content was vigorously swirled. The flask was then placed in the dark for approximately 2 hours 30 minutes. Then, 20 cm<sup>3</sup> of 10% aqueous potassium iodide and 125 cm<sup>3</sup> of water were added using a measuring cylinder. The content was titrated with 0.1 M sodium thiosulphate solution until yellow colour almost disappeared. Few drops of 1% starch indicator was added and titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test. The iodine value was calculated by the expression;

$$I. V = \frac{12.69 \times C \times (V_1 - V_2)}{M} \quad (5)$$

Where,

C = Concentration of sodium thiosulphate



$V_1$  = Volume of sodium thiosulphate used for blank

$V_2$  = Volume of sodium thiosulphate used for determination

M = Mass of sample

### 3.9.4 Free Fatty Acid Value (FAV)

2 g of the extracted oil was weighed into a 250 mL conical flask. 20 cm<sup>3</sup> of ethanol (purity 95%) was added to the flask. The solution was heated slightly at 20°C to aid the dissolution of the fat in the alcohol. 2 drops of phenolphthalein solution was added as indicator.

The obtained yellowish solution was titrated with 0.1 M sodium hydroxide sodium solution while shaking the solution vigorously. The colour of the solution turned pink and at the point when the pink colour persisted for 30 s was termed the end point. The percentage of free fatty acid on the oil (% oleic) was calculated as the expression;

$$\% \text{ FAV} = \frac{V \times N \times 28.2}{\text{weight of sample (g)}} \quad (6)$$

Where,

V = Volume of sodium hydroxide

N = Normality of sodium hydroxide (0.1)

### 3.9.5 Peroxide Value (PV)

10 cm<sup>3</sup> of chloroform was added to 2 g of the sample in a 500 cm<sup>3</sup> conical flask and dissolve quickly by stirring. 15 cm<sup>3</sup> of acetic acid (purity 98%) was added followed by

1 cm<sup>3</sup> freshly prepared saturated potassium iodide solution. The flask was closed and stirred for 1 minute and kept in the dark for 15 minutes, 75 cm<sup>3</sup> of distilled water was added and shaken vigorously. 3 drops of starch solution was added as indicator. The liberated iodine was titrated against 0.01 M sodium thiosulphate. Peroxide value was calculated using the expression;

$$PV = \frac{(V_1 - V_0) \times C \times 1000}{M} \quad (7)$$

Where,

$V_0$  = Volume of sodium thiosulphate

$V_1$  = Volume of sodium thiosulphate for determination

$C$  = Concentration of sodium thiosulphate (0.1 M)

$M$  = Mass of sample

### ***3.10 Soap making by Direct Saponification***

The procedure of direct saponification was followed as Warra et al (2011) and Warra (2012). The direct saponification is saponified with an alkali solution. This works is used two types of the alkali solution in order to produce a solid and liquid soap, which sodium hydroxide (NaOH) and potassium hydroxide (KOH) respectively. However, the base soap of solid and liquid in this work were obtained from the formula that generated from experimental research.

### **3.10.1 Solid soap**

The solid soap was obtained from the sodium hydroxide (as a lye) solution mix with the extracted oils (Warra, 2012). 32.25 gram of sesame oil was poured into 500 cm<sup>3</sup> beaker, followed by 2.15 gram of myristic acid, to add the hardness of the soap. The mixture was slightly heated to dissolve the oil and the myristic acid. Then, 5.10 gram of lye was added into the beaker and swirled vigorously. 10.50 gram of glycerine was added into the mixture and stirred about ten minutes, before it was poured into a mould for drying process. The mould with the mixture was cooled in room temperature for a night before proceed to soap analysis.

### **3.10.2 Liquid soap**

The liquid soap was obtained from the mixture of potassium hydroxide (as a lye) and extracted oils (Aiwize and Achebo, 2012). 12 gram of glycerine was heated until the temperature was up to 40-50°C. Six gram of lye was added into the glycerine and stirred vigorously about ten minutes. Then, 32 gram of sesame oil was added into the mixture and stirred until the mixture is completely mixed for about 1 hour and 30 minutes. The soap formed was diluted with water to the required viscosity and was cooled in room temperature before proceed to soap analysis.

## ***3.11 Soap Composition Analysis***

In this work, the pH value, foam height and observation of colour in solution, odour, foam structure and solubility in water determination are carried out by using the methods reported by Warra et al (2011), Aiwize et al (2012) and Oluwatoyin (2011).

### **3.11.1 The pH Value determination**

The pH was determination using a pH meter. 5 g of solid soap was weighed and dissolved in distilled water in 50 cm<sup>3</sup> volumetric flask. The electrode of the pH meter was inserted into the solution and the reading was recorded.

The steps were repeated by using the liquid soap.

### **3.11.2 The Foam height determination**

0.5 g of solid soap was heated and added to 200 cm<sup>3</sup> measuring cylinder containing 100 cm<sup>3</sup> of distilled water. The mixture was shaken vigorously so as to generate foams. After shaken for about 5 minutes, the cylinder was allowed to stand for about 15 minutes. The height of the foam in the solution was measured and recorded.

The steps were repeated by using the liquid soap.

### **3.11.3 The Observation of the soap**

The observation of solubility in water, colour in solution, foam structure and odour were conducted for both types of soap.

0.5 g of solid soap was mixed with 100 cm<sup>3</sup> of distilled water in a conical flask. The mixture is shaken for about 5 minutes to generate foams, and then was transferred into beaker. The solubility and formation of scum of the soap was observed and recorded. Then, the colour and odour observation also recorded.

The steps were repeated by changing the distilled water to the tap water. After that, the observation is continued by testing the liquid soap. Once completed, the soap was then undergoing a few tests, such as dirty cloth, hand washing and laboratory glassware. The time was taken during hand washing and laboratory glassware testing.

### ***3.12 Soap Composition Testing***

Testing is applied to the dirty cloth, hand washing and laboratory glassware. The observation and timing were recorded.

### ***3.13 Summary***

At the end of this study, all methodologies are successfully tested and done.

## 4 RESULT AND DISCUSSIONS

### *4.1 Overview*

This study were 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, unde-hulled seeds.

This study 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 unde-hulled and dehulled oil, soap will be tested for its availability.

### *4.2 Introduction*

This study presents the result of physiochemical analysis of sesame oil, physiochemical analysis of sesame soap and the characteristic of soap testing.

### *4.3 Physiochemical Analysis of Sesame Oil*

This section shows the result of physiochemical analysis of unde-hulled and dehulled sesame oil. Five parameters were being considered in this experiment, which is free fatty acid value (FFAV), saponification value (SV), acid value (AV), iodine value (IV) and peroxide value (PV). Each parameter was experimented 3 times, in order to judge the accuracy of the analysis value.

#### **A) Free Fatty Acid Value (FFAV)**

The result of free fatty acid experiment of dehulled and unde-hulled oil was shown in Table 4-3A and Table 4-4A, respectively.

Table 4-3A: Data collected for Dehulled Sesame oil (FFAV)

<b>Trial</b>	<b>Dehulled sesame oil, ml</b>		<b>Average</b>
	Start	End	
<b>1</b>	0.00	0.50	0.50
<b>2</b>	0.50	0.90	0.40
<b>3</b>	0.90	1.25	0.35

Table 4-4A: Data collected for Undehulled Sesame oil (FFAV)

<b>Trial</b>	<b>Undehulled sesame oil, ml</b>		<b>Average</b>
	Start	End	
<b>1</b>	0.00	0.35	0.35
<b>2</b>	0.35	0.65	0.30
<b>3</b>	0.65	0.93	0.28

The data collected for dehulled and undehulled sesame oil analysis was tabulated as tables above. The average of 0.1N sodium hydroxide was used for titration in dehulled oil was in range 0.35-0.50 ml. While, for undehulled oil was in range 0.28-0.35 ml. From the data collected, the FFAV was calculated and shown in Appendix A. The lowest titration value was the accurate free fatty acid of the sesame oil, where the reaction was occurred from colourless turned to pink which persisted for 30 seconds.

## B) Saponification Value (SV)

The result for saponification experiment was tabulated as Table 4-5B and Table 4-6B as below for dehulled and unde-hulled sesame oil, respectively.

Table 4-5B: Data collected for Dehulled sesame oil (SV)

Trial	Blank, ml		Dehulled sesame oil, ml	
	Start	End	Start	End
1				52.5
2	0.00	38.7	0.00	52.3
3				52.0

Table 4-6B: Data collected for unde-hulled sesame oil (SV)

Trial	Blank, ml		Unde-hulled sesame oil, ml	
	Start	End	Start	End
1				54.7
2	0.00	38.7	0.00	53.8
3				53.7

As the data shown above, the range of 0.5M HCl was used for titration in dehulled sesame oil was 52.0-52.5 ml and for unde-hulled sesame oil was 53.7-54.7 ml. While for the blank, the titration was used 38.7 ml of 0.5M HCl. The calculation of saponification value was shown in Appendix A. The lowest value calculated was the accurate value for the saponification value, where the pink colour was indicated as the end point.



**C) Acid Value (AV)**

The result of acid experiment for dehulled and unde-hulled sesame oil was tabulated as shown Table 4-7C and Table 4-8C, respectively.

Table 4-7C: Data collected for dehulled sesame oil (AV)

<b>Trial</b>	<b>Dehulled sesame oil, ml</b>		<b>Average</b>
	Start	End	
<b>1</b>	0.00	1.47	1.47
<b>2</b>	1.47	2.93	1.46
<b>3</b>	2.93	4.39	1.46

Table 4-8C: Data collected from unde-hulled sesame oil (AV)

<b>Trial</b>	<b>Undehulled sesame oil, ml</b>		<b>Average</b>
	Start	End	
<b>1</b>	0.00	1.30	1.30
<b>2</b>	1.30	2.55	1.25
<b>3</b>	2.55	3.75	1.20

Data collected in both tables above shows the average volume of N/10 potassium hydroxide (KOH) was used in the titration. For dehulled experiment, the range was in between 1.46-1.47ml, while unde-hulled experiment, the range was in between 1.20-1.30

ml. The acid value was calculated and shown in Appendix A. The lowest value was the accurate acid value for each sample.

#### D) Iodine Value (IV)

The result of iodine experiment was shown in Table 4-9D and Table 4-10D for dehulled and unde-hulled sesame oil, respectively.

Table 4-9D: Data collected for Dehulled sesame oil (IV)

Trial	Blank, ml		Dehulled sesame oil, ml	
	Start	End	Start	End
1				83.5
2	0.00	118.5	0.00	84.0
3				84.0

Table 4-10D: Data collected for Undehulled sesame oil (IV)

Trial	Blank, ml		Undehulled sesame oil, ml	
	Start	End	Start	End
1				84.0
2	0.00	118.5	0.00	84.5
3				85.0

The collected data for acid experiment shows the range of 0.1M sodium thiosulphate was used in titration of Wij's reagent. Wij's reagent was the mixture from iodine

monochloride and glacial acetic acid. For dehulled sesame oil, the range of titration was 83.5-84.0 ml, while undehulled sesame oil was 84.0-85.0 ml. The range shows that the undehulled sesame oil was high than the dehulled titration. The calculation of iodine value was shown in Appendix A. The lowest iodine value was the accurate value for each sample.

### E) Peroxide Value (PV)

Table 4-11E and Table 4-12E show the result of peroxide experiment for dehulled and undehulled sesame oil.

Table 4-11E: Data collected for Dehulled sesame oil (PV)

Trial	Blank, ml		Dehulled sesame oil, ml		Average
	Start	End	Start	End	
1			0.00	3.55	3.55
2	0.00	0.5	3.55	7.05	3.50
3			7.05	10.5	3.45

Table 4-12E: Data collected for Undehulled sesame oil (PV)

Trial	Blank, ml		Undehulled sesame oil, ml		Average
	Start	End	Start	End	
1			0.00	2.20	2.20
2	0.00	0.5	2.20	4.35	2.15
3			4.35	6.45	2.10

Data result shows the average of 0.01M sodium thiosulphate solution was used in titration of peroxide value. The range was in between 3.45-3.55 ml for dehulled, while for undehulled sesame oil was 2.10-2.20 ml. The calculation of peroxide value was shown in Appendix A. The lowest value of peroxide calculated was the accurate peroxide value for each sample.

The overall result from the calculation in Appendix A was tabulated in Table 4-13.

Table 4-13: Overall Value of Physiochemical Analysis of Sesame oil

Parameter	Undehulled	Dehulled
Oil contents (%)	24.38	19
Free Fatty Acid value (% Oleic)	39.48	49.3
Saponification value (mg KOH/g)	210.4	186.5
Acid value (mg KOH/g)	1.34	1.64
Iodine value (g I <sub>2</sub> /100g)	106.3	109.4
Peroxide value (meq H <sub>2</sub> O <sub>2</sub> /g)	8	14.8

Based on Table 4-13 above, sesame seed oil has saponification value of 210.4 mg KOH/ g and 186.5 mg KOH/ g for undehulled and dehulled sesame oil, respectively. According to Warra (2012) and Borchani et. al. (2010), sesame oil for dehulled seed is lower than others saponification value, such as Nigerian cotton seed oil (199.42 mg KOH/ g), Neem seed oil (213 mg KOH/ g), African pear seed oil (246 mg KOH/ g), *Adansonia digitata linn* seed oil (230.01 mg KOH/ g), *Elaeis guineensis* seed oil (246.60 mg KOH/ g), however it is higher than the following oils; *Hyptis spicigera* seed oil (154.10 mg KOH/ g), *Nepoleana imperials* seed oil (77.06 mg KOH/ g), Castor seed oil (123.3 mg KOH/ g), Cashew kernel oil (137.0 mg KOH/ g) and Olive seed oil (97.94 mg KOH/ g). In soap production, high saponification value is justified to produce a hard solid soap. In theoretically, sesame seed oil can produce a hard solid soap.

The iodine value for undehulled and dehulled sesame oil is 106.3 and 109.4 g I<sub>2</sub>/ 100 g, respectively. Compared to the other oils (Warra, 2012; Borchani et. al., 2010), sesame oil is lower than Nigerian cotton seed oil (119.78 g I<sub>2</sub>/ 100 g), however it is higher than olive oil (81.23 g I<sub>2</sub>/ 100 g), Cashew kernel oil (41.3 g I<sub>2</sub>/ 100 g) or Avocado pear oil (42.66 g I<sub>2</sub>/ 100 g). Based on SoapCalc (n.d.), it was claimed that the lower the iodine value, the harder the solid soap can be produced, which below than 70 g I<sub>2</sub>/ 100 g. In theoretically, sesame oil indicates to produce a soft solid soap due to high of iodine value.



Figure 4.4: Sesame Seed Oil

The acid value obtained from the undehulled and dehulled sesame seed oil is 1.34 and 1.64 mg KOH/ g, respectively. The values is higher than olive oil (1.12 mg KOH/ g), but lower than coconut oil (1.866 mg KOH/ g), sunflower oil (1.866 mg KOH/ g) and mustard oil (3.733 mg KOH/ g) (Adithya et. al., 2012; Borchani et. al., 2010). According to Adithya et. al. (2012), lower of acid value indicates that lower of rancidity

of oils. Sesame oil contains of high percentage of free fatty acid, which undehulled (39.48%) and dehulled (49.3%). That means sesame oil has the lower rancidity of oil.

The peroxide value that obtained from undehulled and dehulled sesame seeds oil is 8 meq/kg and 14.8 meq/kg, respectively. The peroxide values obtained is lower than olive oil (20 meq/kg) (Borchani et. al., 2010). According to Borchani et. al. (2010), higher of peroxide value indicates of high level in oxidative rancidity. Therefore, sesame oil is high of stability during extraction due to lower of peroxide value. Figure 4.1.1 above shows the sesame oil that obtained from the extraction, which undehulled (front, right-hand-side) and dehulled (back, left-hand-side) seed oil. Undehulled seed oil is dark yellow and dehulled seed oil is light yellow. As the sesame seeds hull contain a large amount of oxalic acid (Abou-Gharbia et. al., 1997), it cause the oil is darker.

#### ***4.4 Physiochemical Analysis of Sesame Soap***

This section shows the result of physiochemical analysis of soap that produced from sesame oils. The soap was made up from different types of oil, which undehulled and dehulled seed oil with different phases, solid and liquid. There are six parameters that considered, such as foam height, pH value, and observation of odour, colour of the soap in solution, the solubility of soap in water and the foam structure. Table 4-14 shows the data collected during the experiment.

Table 4-14: Physiochemical Analysis of Sesame soap

Parameter	Undehulled (New)		Dehulled	
	Solid	Liquid	Solid	Liquid
Foam height (cm)	5.8	20.0	16.4	25.0
pH	10.48	10.81	10.51	10.54

Odour	Agreeable	Agreeable	Agreeable	Most agreeable
Colour in solution	Slightly cloudy	Yellowish	Cloudy	Light yellow
Solubility in water	Take time to soluble	Soluble	Take time to soluble	Soluble
Foam structure	Creamy	Bubbly	Creamy	Bubbly

For dehulled soap, table above shows the foam height of liquid soap is higher than solid, followed by pH value. For dehulled soap, it also shows the same characteristics of unde-hulled soap, which the foam height of liquid is higher than solid. However, the pH value for both phases, solid and liquid of dehulled soap shows that approximately 10.51 to 10.54, compared to unde-hulled soap, 10.48 and 10.81, respectively. According to Hwang (2005), it found that sesame seed with hull contains a large amount of oxalic acid, meanwhile, sesame without hull will off about 0.25% from the content of oxalic acid. Therefore, the soap becomes less alkali due to the high of acid amount. In addition, pH range of 9-11 was considered as high level of alkali or known as incomplete alkali hydrolysis. The incomplete alkali hydrolysis was happen from the saponification process, due to unfinished heating the oil or superfatting agent with the lye.



The foam height of liquid soap for both seed soap is higher than solid, which 25.0 (dehulled) and 20.0 (unde-hulled) cm. The foam structure for liquid is bubbly, while solid is creamy for both unde-hulled and dehulled soap. The solubility of both soap in water are soluble; however solid soap would take time to completely soluble in water. Solid soap produced a cloudy solution in water, while liquid soap produced a yellowish to light yellow colour in water.

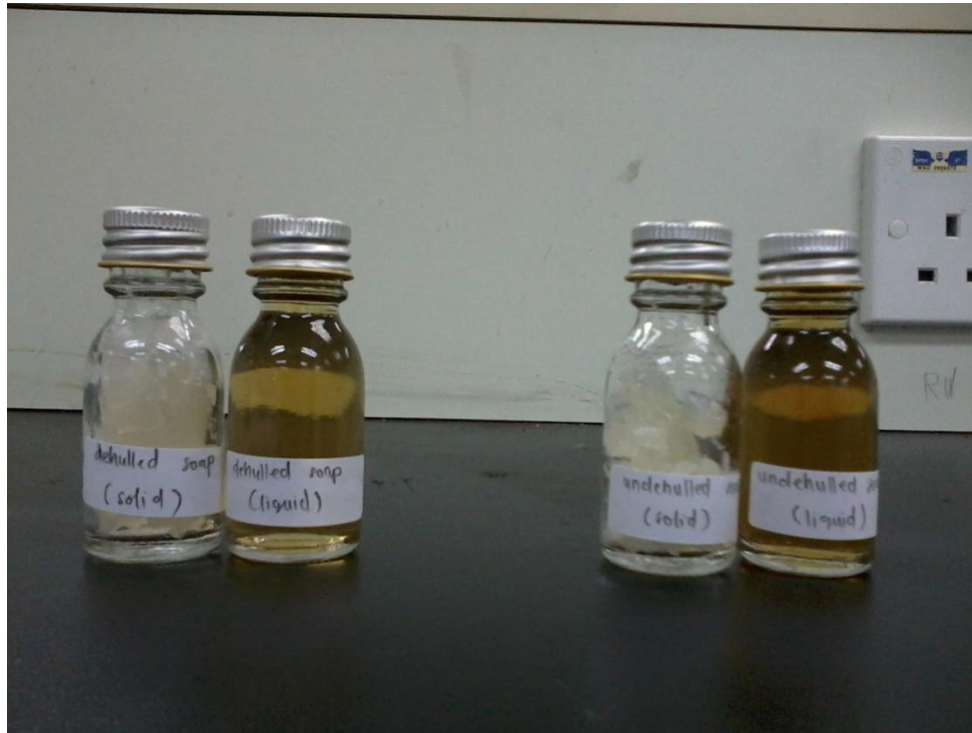


Figure 4.5: Comparison of Sesame oil Soap

From Figure 4.1.2, the difference between undehulled (right) and dehulled (left) soap is the colour of liquid soap, which dehulled soap lighter than undehulled soap. However, the colour of solid soap is considered the same.

As the result obtained from Table 4.1.2, dehulled soap in liquid phase is the most agreeable odour, foam height and pH value. The undehulled soap is also accepted agreeable for some people due to its odour, followed by the solid soaps.

#### 4.5 Sesame oil Soap Testing

In this section will show the result of the sesame oil soap testing. There are three tests in order to fulfil the scope, which hand washing, dirty cloth and glass ware. Table 4-15 shows the data collected during the experiment was done. Time was taken to shows the soap is easy to rinse using a tap water.

Table 4-15: Sesame (*sesamum indicum*) soap Testing

Parameter	Undehulled (New)		Dehulled	
	Solid	Liquid	Solid	Liquid
Hand washing (s)	9	13	15	10
Dirty cloth	Slightly dirt	Clean	Clean	Clean
Glassware (s)	5	5	6	5

Based on Table 4-15, solid soap from undehulled oil (SU) has the fastest time taken for hand washing testing, 9 seconds, while liquid soap from undehulled oil (LU), solid (SD) and liquid (LD) soap from dehulled oil are 13, 15 and 10 seconds, respectively. However, the testing over the dirty cloth for SU show it not clean completely, but slightly dirt; compared to LU, LD and SD shows its clean completely over the dirty cloth testing. Figure 4.1.3a and b in Appendix B shows the comparison before and after the cloths was washed using 4 types of soap. Figure of testing the soap to hand washing and glassware also shows in Figure 4.1.3c and 4.1.3d in Appendix B.

#### **4.6 Summary**

This study present the result that obtained from the sesame seed, where sesame seed , sesame seed were provided 19% of dehulled oil and 24.38% of undehulled oil from 100 gram of seeds. Dehulled seed oil with saponification value of 186.5 mg KOH/ g, acid value of 109.4 g I<sub>2</sub>/100g, peroxide value of 14.8 meq H<sub>2</sub>O<sub>2</sub>/g, and free fatty acid value of 49.3 % (Oleic) is greater value than undehulled seed oil, which 210.4 mg KOH/ g, 1.34 g I<sub>2</sub>/100g, 106.3 meq H<sub>2</sub>O<sub>2</sub>/g, 8 and 39.48 % (Oleic).

The results from Table 4-14 and 4-15; shows that dehulled and undehulled soap have the good characteristics as soap. The range of pH value is acceptable which 10.48, 10.81, 10.51 and 10.54 for solid and liquid of undehulled and dehulled soaps. The odour was also agreeable, especially the liquid dehulled soap.

## 5 CONCLUSIONS

### 5.1 Introduction

Conclusion and recommendations for this study are presented in this chapter. All methodologies, scopes and objectives were successfully tested and produced a yield according to the standard.

### 5.2 Conclusion

This research was proposed to establish the production of soap by dehulled and dehulled sesame seed using soxhlet method. Based on the experiment, sesame seed were provided 19% of dehulled oil and 24.38% of unde-hulled oil from 100 gram of seeds. Dehulled seed oil with saponification value of 186.5 mg KOH/ g, acid value of 109.4 g I<sub>2</sub>/100g, peroxide value of 14.8 meq H<sub>2</sub>O<sub>2</sub>/g, and free fatty acid value of 49.3 % (Oleic) is greater value than unde-hulled seed oil, which 210.4 mg KOH/ g, 1.34 g I<sub>2</sub>/100g, 106.3 meq H<sub>2</sub>O<sub>2</sub>/g, 8 and 39.48 % (Oleic).

The results from Table 4-14 and 4-15; shows that dehulled and unde-hulled soap have the good characteristics as soap. The range of pH value is acceptable which 10.48, 10.81, 10.51 and 10.54 for solid and liquid of unde-hulled and dehulled soaps. The odour was also agreeable, especially the liquid dehulled soap.

In conclusion, the objective of this study is achieved. Sesame seed soap can be established, whether from unde-hulled or dehulled seed.

### 5.3 Future work

The recommendation for improve this study in the future are:

- The saponification process should be longer than 3 hours, in order to improve the alkali hydrolysis.

- Consider more solvent (ethanol and water) in the research to determine the suitable solvent for sesame seed extraction.
- Consider more factors (concentration and ratio of weight) of the ingredient for soap making, to see how the factors affects the soap.

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## **APPENDICES**

**A      RESULT AND CALCULATION OF PHYSICHEMICAL ANALYSIS**

**B      RESULT COLLECTED FROM SESAME OIL SOAP TESTING**

## APPENDIX A

### RESULT AND CALCULATION OF PHYSIOCHEMICAL ANALYSIS

#### Free Fatty Acid (FFAV)

Trial	Dehulled sesame oil		Average
	Start	End	
1	0.00	0.50	0.50
2	0.50	0.90	0.40
3	0.90	1.25	0.35

By using the following formula,

$$\% \text{ FAV} = \frac{V \times N \times 28.2}{\text{weight of sample (g)}}$$

Where,

V = Volume of sodium hydroxide

N = Normality of sodium hydroxide (0.1)

For dehulled sesame oil,

First trial,

$$\begin{aligned} \% \text{ FAV} &= \frac{0.50 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 70.5\% \end{aligned}$$

Second trial,

$$\begin{aligned}\% \text{ FAV} &= \frac{0.40 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 56.4\%\end{aligned}$$

Third trial,

$$\begin{aligned}\% \text{ FAV} &= \frac{0.35 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 49.3\%\end{aligned}$$

From the calculation, the lowest titration is the free fatty acid value, which is 49.3%

<b>Trial</b>	<b>Undehulled sesame oil</b>		<b>Average</b>
	<b>Start</b>	<b>End</b>	
<b>1</b>	0.00	0.35	0.35
<b>2</b>	0.35	0.65	0.30
<b>3</b>	0.65	0.93	0.28

For undehulled sesame oil,

First trial,

$$\begin{aligned}\% \text{ FAV} &= \frac{0.35 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 49.3\%\end{aligned}$$

Second trial,

$$\begin{aligned}\% \text{ FAV} &= \frac{0.30 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 42.3\%\end{aligned}$$

Third trial,

$$\begin{aligned}\% \text{ FAV} &= \frac{0.28 \times 0.1 \times 28.2}{2} \times 100\% \\ &= 39.48\%\end{aligned}$$

Therefore, the lowest value of the free fatty acid is 39.48%

### Saponification Value (SV)

Trial	Blank, ml		Dehulled sesame oil, ml	
	Start	End	Start	End
1				52.5
2	0.00	38.7	0.00	52.3
3				52.0

By using the following formula,

$$S.V = \frac{(S - B) \times M \times 56.1}{\text{sample weight (g)}}$$

Where,

S = Sample titrate value

B = Blank titrate value

M = Molarity of the HCl

56.1 = Molecular weight of KOH

For dehulled sesame oil,

First trial,

$$S.V = \frac{(52.5 - 38.7) \times 0.5 \times 56.1}{2}$$

$$= 193.5 \text{ mg KOH/ g}$$

Second trial,

$$S.V = \frac{(52.3-38.7) \times 0.5 \times 56.1}{2}$$
$$= 190.7 \text{ mg KOH/ g}$$

Third trial,

$$S.V = \frac{(52.0-38.7) \times 0.5 \times 56.1}{2}$$
$$= 186.5 \text{ mg KOH/ g}$$

Therefore, the saponification value of dehulled sesame oil is 186.5 mg KOH/ g

Trial	Blank, ml		Undehulled sesame oil, ml	
	Start	End	Start	End
1				54.7
2	0.00	38.7	0.00	53.8
3				53.7

For undehulled sesame oil,

First trial,

$$S.V = \frac{(54.7-38.7) \times 0.5 \times 56.1}{2}$$
$$= 224 \text{ mg KOH/ g}$$



Second trial,

$$S.V = \frac{(53.8-38.7) \times 0.5 \times 56.1}{2}$$
$$= 211.8 \text{ mg KOH/ g}$$

Third trial,

$$S.V = \frac{(53.7-38.7) \times 0.5 \times 56.1}{2}$$
$$= 210.4 \text{ mg KOH/ g}$$

Therefore, the saponification value of undehulled sesame oil is 210.4 mg KOH/ g

### Acid Value (AV)

Trial	Dehulled sesame oil, ml		Average
	Start	End	
1	0.00	1.47	1.47
2	1.47	2.93	1.46
3	2.93	4.39	1.46

By using the following formula,

$$A.V = \frac{5.61 \times \text{no. of mL } 0.1 \text{ M KOH}}{\text{weight, g}}$$

For dehulled sesame oil,

First trial,

$$A.V = \frac{5.61 \times 1.47}{5}$$
$$= 1.65 \text{ mg KOH/ g}$$

Second trial,

$$A.V = \frac{5.61 \times 1.46}{5}$$
$$= 1.64 \text{ mg KOH/ g}$$

Third trial was the same as the second trial, since the value was same, 1.46 ml.

Therefore, the acid value for dehulled sesame oil is 1.64 mg KOH/g.

Trial	Undehulled sesame oil, ml		Average
	Start	End	
1	0.00	1.30	1.30
2	1.30	2.55	1.25
3	2.55	3.75	1.20

For undehulled sesame oil,

First trial,

$$\begin{aligned} \text{A.V} &= \frac{5.61 \times 1.30}{5} \\ &= 1.46 \text{ mg KOH/ g} \end{aligned}$$

Second trial,

$$\begin{aligned} \text{A.V} &= \frac{5.61 \times 1.25}{5} \\ &= 1.40 \text{ mg KOH/ g} \end{aligned}$$

Third trial,

$$\begin{aligned} \text{A.V} &= \frac{5.61 \times 1.20}{5} \\ &= 1.34 \text{ mg KOH/ g} \end{aligned}$$

Therefore, the acid value for undehulled sesame oil is 1.34 mg KOH/ g

### Iodine Value (IV)

Trial	Blank, ml		Dehulled sesame oil, ml	
	Start	End	Start	End
1				83.5
2	0.00	118.5	0.00	84.0
3				84.0

By using the following formula,

$$I. V = \frac{12.69 \times C \times (V_1 - V_2)}{M}$$

Where,

C = Concentration of sodium thiosulphate

V<sub>1</sub> = Volume of sodium thiosulphate used for blank

V<sub>2</sub> = Volume of sodium thiosulphate used for determination

M = Mass of sample

For dehulled sesame oil,

First trial,

$$\begin{aligned} I. V &= \frac{12.69 \times 0.1 \times (118.5 - 83.5)}{0.4} \\ &= 111.0 \text{ g I}_2 / 100\text{g} \end{aligned}$$

Second trial,

$$\text{I. V} = \frac{12.69 \times 0.1 \times (118.5 - 84.0)}{0.4}$$
$$= 109.4 \text{ g I}_2 / 100\text{g}$$

Third trial,

$$\text{I. V} = \frac{12.69 \times 0.1 \times (118.5 - 84.0)}{0.4}$$
$$= 109.4 \text{ g I}_2 / 100\text{g}$$

Therefore, the iodine value for dehulled sesame oil is 109.4 g I<sub>2</sub> / 100g

Trial	Blank, ml		Undehulled sesame oil, ml	
	Start	End	Start	End
1				84.0
2	0.00	118.5	0.00	84.5
3				85.0

For undehulled sesame oil,

First trial,

$$\begin{aligned} \text{I. V} &= \frac{12.69 \times 0.1 \times (118.5 - 84.0)}{0.4} \\ &= 109.4 \text{ g I}_2 / 100\text{g} \end{aligned}$$

Second trial,

$$\begin{aligned} \text{I. V} &= \frac{12.69 \times 0.1 \times (118.5 - 84.5)}{0.4} \\ &= 107.8 \text{ g I}_2 / 100\text{g} \end{aligned}$$

Third trial,

$$\begin{aligned} \text{I. V} &= \frac{12.69 \times 0.1 \times (118.5 - 85.0)}{0.4} \\ &= 106.3 \text{ g I}_2 / 100\text{g} \end{aligned}$$

Therefore, the iodine value for undehulled sesame oil is 106.3 g I<sub>2</sub> / 100g

### Peroxide Value (PV)

Trial	Blank, ml		Dehulled sesame oil, ml		Average
	Start	End	Start	End	
1			0.00	3.55	3.55
2	0.00	0.5	3.55	7.05	3.50
3			7.05	10.5	3.45

By using the following formula,

$$PV = \frac{(V_1 - V_0) \times C \times 1000}{M}$$

Where,

$V_0$  = Volume of sodium thiosulphate

$V_1$  = Volume of sodium thiosulphate for determination

C = Concentration of sodium thiosulphate (0.1 M)

M = Mass of sample

For dehulled sesame oil,

First trial,

$$PV = \frac{(3.55 - 0.5) \times 0.01 \times 1000}{2}$$
$$= 15.3 \text{ meq H}_2\text{O}_2 / \text{g}$$

Second trial,

$$PV = \frac{(3.50-0.5) \times 0.01 \times 1000}{2}$$
$$= 15 \text{ meq H}_2\text{O}_2/\text{g}$$

Third trial,

$$PV = \frac{(3.45-0.5) \times 0.01 \times 1000}{2}$$
$$= 14.8 \text{ meq H}_2\text{O}_2/\text{g}$$

Therefore, the peroxide value for dehulled sesame oil is 14.8 meq H<sub>2</sub>O<sub>2</sub>/g

Trial	Blank, ml		Undehulled sesame oil, ml		Average
	Start	End	Start	End	
1			0.00	2.20	2.20
2	0.00	0.5	2.20	4.35	2.15
3			4.35	6.45	2.10

For undehulled sesame oil,

First trial,

$$PV = \frac{(2.20-0.5) \times 0.01 \times 1000}{2}$$
$$= 8.5 \text{ meq H}_2\text{O}_2/\text{g}$$



Second trial,

$$\text{PV} = \frac{(2.15-0.5) \times 0.01 \times 1000}{2}$$
$$= 8.25 \text{ meq H}_2\text{O}_2 / \text{g}$$

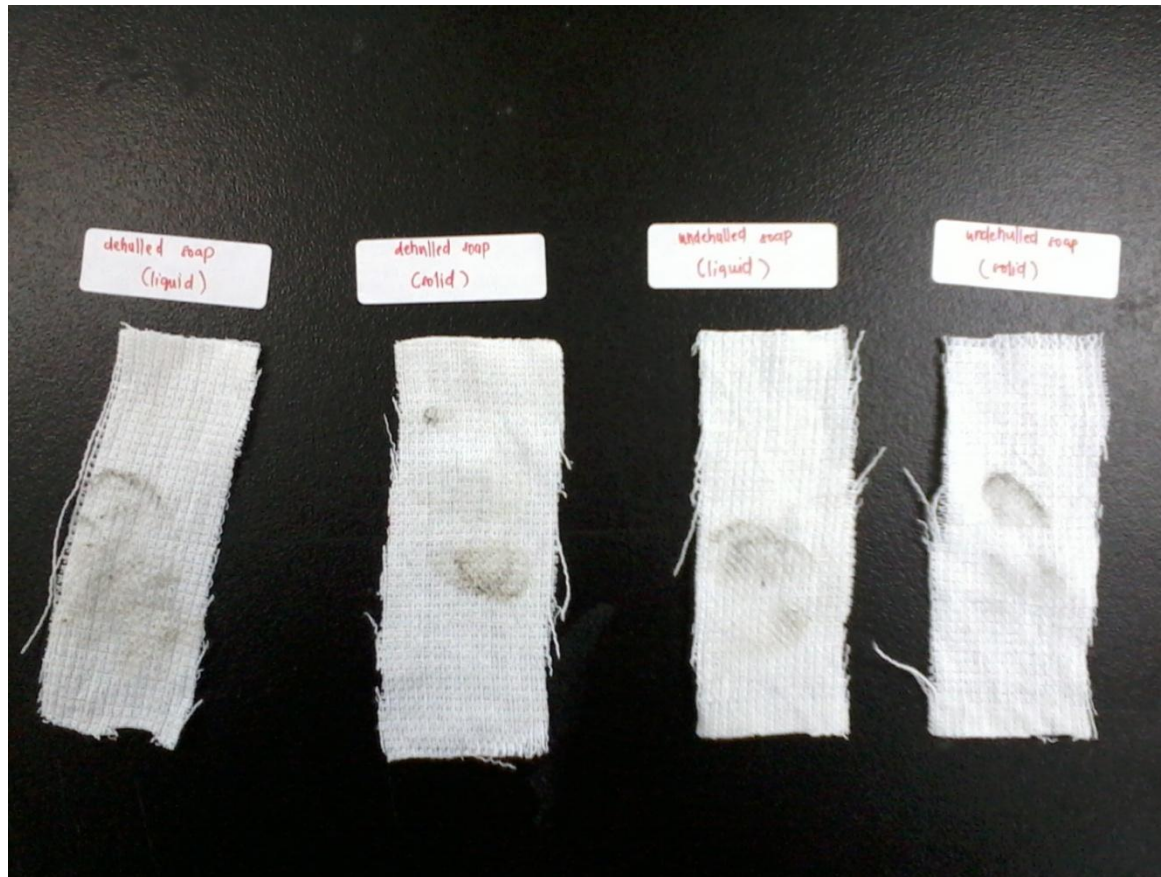
Third trial,

$$\text{PV} = \frac{(2.10-0.5) \times 0.01 \times 1000}{2}$$
$$= 8 \text{ meq H}_2\text{O}_2 / \text{g}$$

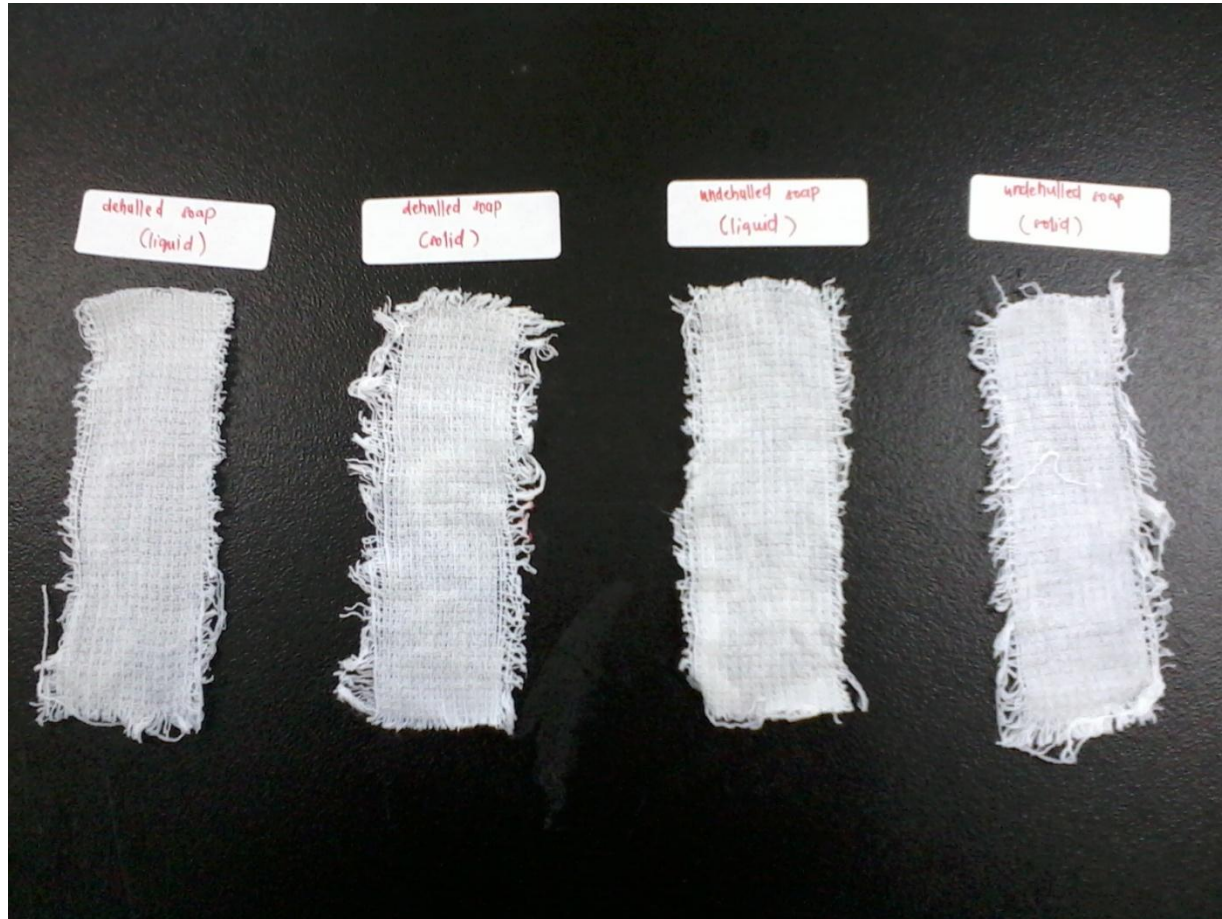
Therefore, the peroxide value for dehulled sesame oil is 8 meq H<sub>2</sub>O<sub>2</sub> / g.

**APPENDIX B**

**RESULT COLLECTED FROM SESAME OIL SOAP TESTING**



**Figure 4.1.3a: Dirty Cloth Testing (Before)**



**Figure 4.1.3b: Dirty Cloth Testing (After)**



**Figure 4.1.3c: Hand Washing Testing**



**Figure 4.1.3d: Glassware Testing**