

CHARACTERIZATION AND MODIFICATION
OF CASTOR OIL EXTRACTED FROM THE
NEWLY MALAYSIAN PRODUCED CASTOR
BEANS

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FROM THE NEWLY MALAYSIAN PRODUCED CASTOR BEANS

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SUPERVISOR'S DECLARATION

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 degree of Bachelor in Chemical Engineering.

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STUDENT'S DECLARATION

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

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ABSTRACT

Vegetable oils, either edible or non-edible, are widely used in huge number of applications in industry. *Ricinus communis* (castor plant) is one of the non-edible vegetable oil and its usage in many industries as a raw material and additives is rapidly growing. In this research, the Malaysian newly produces castor beans are extracted and the castor oil is characterized. The castor oil that is obtained from extraction is refined and modified for further uses. The extraction process involve four steps which are clearing, drying, winnowing and also grinding to get high yield of oil. The castor cake then extracted using the soxhlet extractor and hexane as the solvent. The extracted castor oil produced is characterize by determining the acid value, saponification value, iodine value, specific gravity, viscosity of the oil, the refractive index, and pH value. The crude oil from the extraction process then refines by degumming and neutralization process and then the neutralize oil is bleached. The Malaysian castor oil properties are compared with the ASTM standard specifications. The conclusion is the properties of Malaysian crude and refined castor oil is determined and be compared with the world standard specification of castor oil. The refined castor oil then modified for further uses. The different castor seed from different area which is Casa 5 and Casa 101 is extracted to obtain the castor oil and identify which location in Malaysia that the castor seed produces more oil content and have the same standard with world castor oil quality.

ABSTRAK

Minyak sayuran, samada boleh dimakan atau tidak boleh dimakan telah banyak digunakan dalam pelbagai kegunaan di dalam industri. Pokok castor adalah sejenis minyak sayuran yang tidak boleh dimakan dan banyak digunakan di dalam pelbagai industri sebagai bahan mentah dan bahan tambahan. Dari penyelidikan ini, biji castor yang baru dihasilkan di Malaysia diekstrak dan di sifatnya fizikal dan kimianya di kaji. Proses mengekstrak minyak castor di mulakan dengan membersihkan, mengeringkan, mengupas dan menghancurkan biji castor untuk memperoleh peratusan minyak yang tinggi. Biji castor yang hancur diekstrak menggunakan pengekstrak 'Soxhlet' dan hexane sebagai pelarut. Minyak castor yang di peroleh dari biji castor yang diekstrak di tapis dan di ubahsuai untuk kegunaan lain. Minyak castor yang diperolehi di kaji kualitinya dari segi nilai asid, nilai iodin, nilai kelikatan, nilai index biasan, nilai pH, nilai SG(specific gravity) dan nilai SV(saponification value). Minyak mentah kemudiannya di tapis dengan proses penyah-gam(degumming), di netralkan dan dilunturkan. Sifat minyak castor di bandingkan dengan ASTM kualiti. Kesimpulannya, minyak mentah dan minyak ditapis dan dituliskan di banding sifatnya dengan kualiti minyak castor antarabangsa. Minyak yang ditapis di ubahsuai untuk kegunaan lain. Biji castor yang ditanam di kawasan yang berbeza diekstrak dan mengenalpasti kawasan mana di Malaysia yang menghasilkan biji castor yang dapat mengeluarkan banyak minyak dan mempunyai kualiti minyak setaraf dengan kualiti minyak castor dunia.

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CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

The Vegetable fats or vegetable oils have an essential function in the industrial economy of a developing country as the seed oil provide a huge use in human daily life in order to complete and make the nowadays life more easier. The seed oils are one of the vegetable oil family members. Vegetable oils or vegetable fats are the lipid materials that been derived from the natural plants which physically oil are in liquid state in the room temperature whereas the fat exists in solid state in the room temperature (Ndiaye *et. al.*, 2006). The vegetable oil is composed of triglycerides which lack glycerin in its structure.

Oils that are extracted from plants have been used in this world since the ancient times and already used in many cultures. As an example the castor plant has been known to man for ages. Castor beans have been found in ancient Egyptian tombs dating back to 4000 B.C and during that time, the castor oil was used thousands of years ago in the wick lamps for lighting (Scarpa *et. al.*, 1982). Basically, to obtain the oil from the plants or seeds, the seeds and the plants are through extraction process and then be distillate to remove the solvent used as to separate the extracted oil and solvent used in order to get the pure oil (Kirk-Othmer, 1979).

Several feedstocks from vegetable source such as soybean, rape seed, canola, palm, corn, Jatropha and castor seeds have been studied as an alternative to oil candidate. Among these sources, castor seeds are a potentially promising feedstock since among vegetable oils, castor oil is distinguished by its high content (over 85%)

of ricinoleic acid. There is no other vegetable oil contains so high a proportion of fatty hydroxyacids and castor oil is the most stable viscosity of any vegetable oil (Ogunniyi, 2006).

There are variety processes or the combination of the processes to obtain the oils from the castor seeds. The hydrate presses, continuous screw presses and also solvent extraction are the common methods to obtain the oils from the castor seeds. However, the most satisfactory approach to get the oil is hot pressing the castor seeds by using a hydraulic press and then followed by solvent extraction. This proposal is however focused at extraction of castor oils using solvent. The castor seed firstly face the separation process of the shell from the nibs and then by using the mortar and pestle, the seeds were crushed into the paste (cake) to release the castor fat for the extraction process (Ogunniyi, 2006).

1.2 IDENTIFICATION OF PROBLEM

Currently, the leading producers and the countries that seriously involve in the production of castor oils are India, China, and Brazil. Together, these countries account for 90% of the acreage and production of castor beans. It is grown in Costa Rica, Ecuador, Thailand, Philippines, Paraguay, Romania, Sudan, Mexico, Pakistan, Ethiopia, and Tanzania. The world-wide production stood at 1, 227, 669 tonnes in 2000 (FAO). However, India is the world's largest producer of castor seeds and oils that meets most of global demand for castor oil. India contributes about 750, 000 tonnes annually, and accounting for 60% of the entire global production. Essentially, all the castor oil production in the U.S has been eliminated by a combination of economic factors, excessive allergenic reactions of field and the processing workers, and the toxicity of the seed meal. The toxic inside the castor seed which is known as ricin, is a very dangerous to human as it can kill adults if two or three castor seeds were chewed (Ogunniyi, 2006).

In Malaysia, the castor seeds have been planted in order to fulfill the demand for castor seeds. Casa Kinabalu Sdn.Bhd. is the company that is responsible in the production and the planting the castor plant in Malaysia. This company still new in Malaysia and the first castor seeds that harvest in Malaysia was at the end of

October 2010. Casa Kinabalu Sdn.Bhd already plant the castor beans about 7000 acres in Lundu, Sarawak, 30 acres in Gua Musang, 10 acres in Lukut, Negeri Sembilan, 5 acres in Muar, 10 acres in Kulai, Johor, and also 20 acres in Kedah. Unfortunately, all the castor beans that obtain are exported to the China. Based on the situation happen, we are proposing to extract the newly Malaysian castor seeds in order to obtain the oil that the oils than be characterize and compare the physical and chemical properties of Malaysian castor oil with global castor oil properties. The castor oil extracted than will react with sulphuric acid in process called sulphation to modify the refined castor oil (Ogunniyi, 2006) .

1.3 STATEMENT OF OBJECTIVES

The main objectives of this study are to extract the castor oil by using the different extraction method, characterize the crude and refined castor oil that then be modified using sulfation method for newly Malaysian castor bean.

1.4 RESEARCH SCOPES

The research scopes for this study are:

- i. To extract the castor oil from Malaysian castor seed by using soxhlet extractor and solid-liquid extraction to compare the product percentage oil.
- ii. To characterize the crude and the refined castor oil for easy identification and also assess it quality by combining the extracted oil quality with the national castor oil standard.
- iii. To determine either the Soxhlet extraction process or Solid-Liquid extraction can give more percentage of oil
- iv. To extract the crushed castor seed(kernel together with husk) and compared the oil obtained with dehuled castor seed(kernel only)
- v. To modified the refined castor oil for another uses.

1.5 RATIONALE AND SIGNIFICANCES OF STUDY

Due to the importance of the vegetable oils in the industrial, pharmaceutical, food industries, and also medical, there is an urgent need to produce more oil from the natural plant. In view of this, castor oil is a promising vegetable oil because it has several advantages; it is renewable, environmental friendly and produce easily in the rural areas, where there is an acute need for modern forms of energy. The primary use of the castor oils is as the basic ingredient in the production of nylon 11, sebacic acid, plasticizers and engine jet lubricant. Castor oil's high lubricity which reduces the friction is superior to other vegetable oils and petroleum-based lubricants. It is really clings to metal, especially hot metal, and the castor oils is used in production nylon 6-10, heavy duty automotive greases, coating and inks, surfactants, polyurethanes, soaps, polishes, synthetic resins, fibers, paints, varnishes, dyes, leather treatments, hydraulic fluids and also sealants(Ogunniyi, 2006). Specification for pharmaceutical use can be found in the European Pharmacopoeia. The industrial type maybe divided into three types of quality. 'First' quality is the oil that obtains from only one pressed castor oil and extracted without solvent. This kind of oil normally produced in Europe, is virtually colorless and has very low acidity. 'Second' and 'third' quality of castor oil is commercial names, meaning that the oil has been extracted using solvent (Ogunniyi, 2006).

The oils from the castor seed are very well known as a laxative and purgative that has been widely used for over 2, 000 years. This oil is so effective that it is regularly used to clear the digestive tract in cases of poisoning. The castor oil also has a remarkable antidandruff effect. It is well tolerated by the skin and so is sometimes used as a vehicle for medicinal and cosmetic preparations. When the alcoholic solution is distilled in the presence of sodium salts of higher fatty acids, castor oil congeals to a gel-mass. This useful gel is used in the treatment of dermatosis and is good protective in cases of occupational eczemas and dermatitis. Medical applications consume a tiny fraction of total production. The castor seeds and residual cake are highly poisonous and unless processed to remove the poisons cannot be fed to livestock (Anandan *et. al.*, 2005). In some countries, castor cake is

used as a fertilizer. The poisons or the toxics that contain in the castor cake include ricin (Burdock *et. al.*, 2006).

CHAPTER 2

LITERATURE REVIEW

2.1 BASIC FACTS ABOUT CASTOR OIL

The trade in castor oil as an item of commerce goes back to antiquity (Ogunniyi, 2006). The castor oil is obtained from the extracting method or pressing the castor seed which known with botanical name *Ricinus Communis*. This oil is inexpensive, environmentally friendly and also a naturally-occurring resource. The oil that comes from castor is a viscous, pale yellow color, non-volatile, and also non-drying oil with a bland taste and sometimes used as a purgative. Furthermore, the oil has a slight characteristic odour while the crude oil tastes slightly acrid with a nauseating after-taste. The oil is one of the relative in the vegetable oil family which has a good shelf life and it does not turn rancid unless the oil subjected to excessive heat. The largest exporter of castor oil is India and the other major producers of castor oil are China and Brazil as shown Table 2.1. The total world production of the extracted castor oil is about 500, 000 tonnes and the production of seeds are estimated at one million tonnes (Ogunniyi, 2006).

Table 2.1: Production volume of Castor Oil by major producers

Production volume of castor oil by major producers ^a											
Major producers	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999	2000
	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t	'000 t
India	192	239	232	242	271	333	344	278	304	294	324
China, PR	77	86	93	97	97	82	73	83	80	91	105
Brazil	77	73	54	28	28	22	21	43	21	19	52
Thailand	18	18	19	18	16	14	10	9	9	7	5
E.U. ^b	20	16	14	12	14	11	9	10	7	8	8
Others	54	52	49	41	20	21	22	19	20	23	23
Total	438	484	461	438	446	483	479	442	441	442	517

^a Source—<http://www.ciara.com.ar/estadize.htm>.

^b E.U.—European Union.

Source: Ogunniyi, (2006)

2.1.1 The Castor Oil Properties

Relative to other vegetable oils, castor oil has different physical and chemical properties which vary with the method of extraction the oil. The castor oil that obtain from the cold pressing has low acid value with low iodine value and has slightly higher saponification value compared to the solvent-extracted oil, and the oil is lighter in color (Ogunniyi, 2006). The typical properties are shown in the table 2.2 and the representative composition of oil is given in figure 2.1. The chemistry of castor oil is focused on its high content of ricinoleic acid and also the other three points of function that exist in the castor molecule. One of the molecule functions is the carboxyl group that can give a huge and wide range of the esterifications. Second one is the single points of unsaturation which can be altered using the hydrogenation process or the epoxidation process or the vulcanization process. The last one is the hydroxyl group in the castor oil can be acetylated or alkoxyated maybe can be remove from the oil molecule by using the dehydration process to increase the unsaturation of the oil compound to provide the semi-drying castor oil. By high-temperature pyrolysis and by caustic fusion, the hydroxyl position of oil which is so reactive the molecule can be split at that point to yield useful product with shorter chain length. Actually, the presence of the hydroxyl group on the castor oil is adding the extra stability to the castor oil and also preventing the formation of hydroperoxides.

Table 2.2: Characteristics of castor oil in different situation
Characteristics of castor oil grades

Properties	Cold-pressed oil	Solvent-extracted oil	Dehydrated oil
Specific gravity	0.961–0.963	0.957–0.963	0.926–0.937
Acid value	3	10	6
Iodine value (W_{ij})	82–88	80–88	125–145
Saponification value	179–185	177–182	185–188

Source: Ogunniyi, (2006)



R^I = other fatty acid derivatives

Figure 2.1: Equation showing the constitution of castor oil (Ogunniyi, 2006)

The ricinoleic acid that exists inside the castor oil comprises over 89% of the fatty acid of the oil. The other fatty acids that present are linoleic acid (4.2%), oleic acid (3.0%), stearic acid (1%), palmitic acid (1%), dihydroxystearic acid (0.7%), linolenic acid (0.3%), and eicosanoic acid (0.3%) (G. R. O'Shea Company).

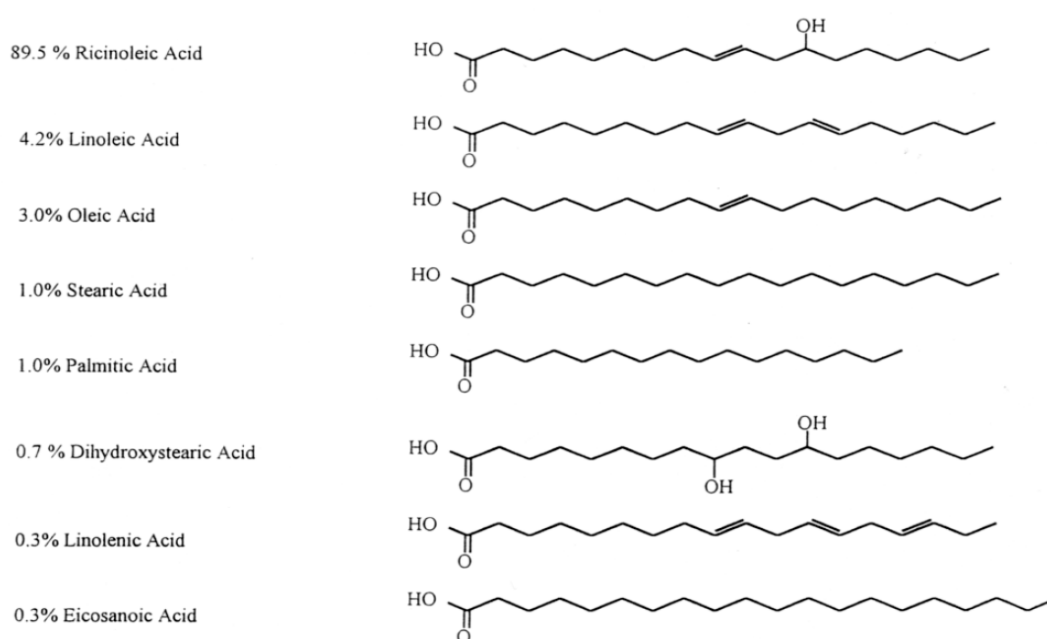


Figure 2.2: Composition of castor oil fatty acids

The castor oil consists mainly of ester of 12-hydroxy-9-octadecenoic acid (ricinoleic acid) as the present of hydroxyl groups and the double bonds makes the oil suitable for many chemical reaction and modification. Various reaction of castor oil is given in table 2.3. Furthermore, the oil is characterized by high viscosity although this is unusual for a natural vegetable oil (Turner *et. al.*, 2004). This behavior is due largely to hydrogen bonding of its hydroxyl groups. The castor oil is soluble in the alcohols in any proportion. However, this oil is limited solubility in aliphatic petroleum solvents. Castor oil is a unique naturally-occurring polyhydroxy compound that has the limitation which is the hydroxyl value and acid value is reducing on the storage. The acid value and hydroxyl value may change about 10% if the oil is stored for about 90 days. The reason why this situation happened is the reaction between the hydroxyl and carboxyl group of oil molecule to form estolides.

Table 2.3: Production a variety of derivatives from chemical reaction

	Nature of Reaction	Added Reactants	Type of Products
Ester Linkage	Hydrolysis	Acid, enzyme, or Twitchell reagent catalyst	Fatty acids, glycerol
	Esterification	Monohydric alcohols	Esters
	Alcoholysis	Glycerol, glycols, pentaerythritol, etc.	Mono- and diglycerides, monoglycols, etc.
	Saponification	Alkalies, alkalies plus metallic salts	Soluble soaps, insoluble soaps
	Reduction	Na reduction	Alcohols
	Amidation	Alkyl amines, alkanolamines, etc.	Amine salts, amides
	Halogenation	SOCl ₂	Fatty Acid halogens
Double Bond	Oxidation, Polymerization	Heat, oxygen, crosslink agent	Polymerized oils
	Hydrogenation	Hydrogen (moderate pressure)	Hydroxystearates
	Epoxidation	Hydrogen peroxide	Epoxidized oils
	Halogenation	Cl ₂ , Br ₂ , I ₂	Halogenated oils
	Addition Reactions	S, maleic acid	Polymerized oils
	Sulfonation	H ₂ SO ₄	Sulfonated oils
Hydroxyl Group	Dehydration, Hydrolysis, distillation	Catalyst (plus heat)	Dehydrated castor oil, Octadecadienoic acid
	Caustic fusion	NaOH	Sebacic acid, capryl alcohol
	Pyrolysis	High heat	Undecylenic acid, heptaldehyde
	Halogenation	PCl ₃ , POCl ₃	Halogenated castor oils
	Alkoxylation	Ethylene and/or propylene oxide	Alkoxyated castor oils
	Esterification	Acetic-, phosphoric-, maleic-, phthalic anhydrides	Alkyl and alkylaryl esters, phosphate esters
	Sulfation	H ₂ SO ₄	Sulfated castor oil (Turkey red oil)
	Urethane reactions	Isocyanates	Urethane polymers

Source: Ogunniyi, (2006)

2.2 FEATURES OF CASTOR SEEDS

The castor plant grows in the wild in large quantities in most tropical and sub-tropical countries. The plant is available at the low price and it is known to tolerate in varying weather condition. Normally, the castor plant needs a temperature between 20 and 26 °C with low humidity throughout the growing season in order to give maximum yield. Moreover, the weather conditions for its growth limit its cultivation to tropical areas of the developing world. There are different types of castor seeds all around the world but on the average, the castor seeds contain about 30 to 55% of oil by weight. The seeds are very poisonous to human and also animals as the seeds contain ricin, ricinine and certain allergens which are toxics. The effects if the castor seed is accidentally ingested, the victims will face abdominal pain, vomiting and diarrhea and as little as 1 mg of the ricin can bring the human to death. The fear of accidental ingestion of the poisonous castor seed by the children does not encourage the use of the castor plant for ornamental purpose. Besides that, the seed cake is poisonous and consequently not suitable for animal feed. Some people who worked with the meal highly develop allergic reaction such as asthma. The main reason why the US farmers no longer grow the castor plant is because of the toxicity of castor seed extensively. However, the pure castor oil if used in right and recommended quantities can be used as the laxative. The quality of the seed oil is hardly affected by the variation in good or poor seeds. The oil from the castor seed is non-edible oil which can be used and can free up some edible oils used in industries for human consumption. The castor plant and castor seed are shown in figure 2.3 and figure 2.4.



Figure 2.3: The castor plant



Figure 2.4: The castor seed

Source: Ogunniyi, (2006)

2.2.1 Extraction of Castor Seed Oil

There are many ways to obtain the oil from the castor seeds. The extraction of oil from the castor seed is one or combination of processes, such as mechanical pressing and solvent extraction. The examples of the mechanical pressing are hydrate presses and continuous screw presses. However, the most effective way to obtain the oil from the seed is hot pressing using a hydraulic press and followed by solvent extraction (Akpan *et. al.*, 2004). In the mechanical pressing, the seeds are crushed to remove the seed from the shell and the adjusted to low the moisture content by warming in a steam-jacketed vessel or in the oven. After that, the crushed seeds are placed into the hydraulic presses and the seeds are pressed by until become the cake to extract the oil. The properties of oil from the mechanical pressing are the oil has the light colour and low free fatty acids (Ogunniyi, 2006). However, about 45% of oil present by the mechanical pressing and the remaining oil in the castor cake can be recovered only by the solvent extraction method. During extraction method using solvents such as heptanes, hexane and petroleum ethers, the seed cakes are extracted with the solvent in the Soxhlet extractor or the commercial extractor.

2.2.2 Refining the Castor Oil

As in other vegetable oils, it is a usual process to refine the crude oil that obtained from either mechanical pressing or solvent extraction. The main objective of refining is to remove the impurities like colloidal matter, free fatty acid, colouring matter and other undesirable constituents, thus making the oil more resistant to deterioration during storage. Refining process includes several steps. Firstly, the solid and colloidal matters are removed by settling and filtration. Then, free fatty acid of the oil is neutralizing by using the alkali. The coloured matter is removes by bleaching process and lastly, the oil will face the deodorizing step by using the treatment with steam at high temperature and low pressure. The standard method of refining the edible oil is applicable to the castor oil.

2.3 USES OF CASTOR OIL

Although the castor oil is not edible oil, it is more versatile than other vegetable oils as the castor oil is widely used as a starting material for many industrial chemical products because of its unique structure. The castor oil is one of those vegetable oils that have found usage in many chemical industries. It is the raw material for many chemical products and also as the additives in drugs. The usage of castor oil can be divided into industrial, food, medicine and also biodiesel and biofuel industries.

2.3.1 Industrial castor oil

As the castor oil is non-edible oil, it is very useful to replace the edible that used in industries. The castor oil has numerous uses in transportation, cosmetics and pharmaceutical and manufacturing industries. For example, the castor oil is used as the adhesives(Yeadon *et. al.*, 1959), brake and hydraulic fluids (Shough, 1942), caulks, dyes, electrical liquid dielectrics, lubricating greases, machining oils, paints(Nezihe, *et. al.*, 2011), pigments, polyurethane adhesives(Kirk-Othmer, 1979), refrigeration lubricant, washing powder, sealants, textiles, waxes, leather treatment, and lacquers. The vegetable oils are very interesting due to their good lubricity and biodegradability as they are attractive alternatives for petroleum-derived lubricants, but the vegetable oils has the weakness as oxidative stability and low temperature performance limits their widespread uses. Not like the other natural vegetable oils, the castor oil has better low temperature viscosity properties and also high temperature lubrication than most of vegetable oils. So, the castor oil is very useful as the lubricant in jet, diesel and race car engines. Castor oil also used in the industries as the raw material for the production of a number of chemicals such as notably sebacic acid which is used in plasticizer and manufacture of dioctyl sebacate (a jet lubricant), undecylenic acid (perfume formulation) (Das *et. al.*, 1989) and also nylon-11.

2.3.2 Castor oil in food

Castor oil is used in the food industry as the food additives, flavourings, candy like chocolate, as a mold inhibitor and also in packaging. The castor oil is known as the non-edible oil which means the castor oil is not safe to eat. By using the cold press process, the castor oil that obtain is safe to eat as the cold press oil contain low acidic and iodine value (Burdock *et. al.*, 2006). The polyoxyethylated castor oil is also used in the foodstuff industries.

2.3.3 Medicinal use of castor oil

The castor oil is categorized as ‘generally recognized as safe as effective’ (GRASE) by the United States Food and Drug Administration (FDA), for over the counter use as a laxative, with its major site of action the small intestine. However, although it may be used for constipation, it is not a preferred treatment. Undecylenic acid, a castor oil derivative, is also FDA approved for over the counter use on skin disorders or skin problems. Nowadays, the modern drugs are rarely given in a pure chemical state, so, the most active content of the medicine combined with the additives. The castor oil or the castor oil derivative is added to many modern drugs. The examples are Miconazole (an anti-fungal agent), Paclitaxel (a mitotic inhibitor used in cancer chemotherapy), Nelfinavir mesylate (an HIV protease inhibitor), Xenaderm ointment (a topical treatment for skin ulcers), and Aci-jel (used to maintain the acidity of the vagina).

2.3.4 Castor oil as Biodiesel and Biofuel

The extracted castor oil which is called the crude castor oil, is refined and the biofuel and biodiesel is produced through the process of esterification and transesterification (Canoira *et. al.*, 2010). The transesterification process is hugely eliminates the tendency of the castor oils and fats to undergo the polymerization and auto-oxidation process. This process also reduces the viscosity of the castor oil so that the castor oil viscosity about the same as the petroleum diesel viscosity (Valente *et. al.*, 2011).

2.3.5 Castor Meal

The castor meal or the castor cake waste is the excellent bio-fertilizer as it contains optimal composition of nutrients especially N-P-K. The toxic called ricin in the castor meal makes the castor meal unsuitable for animal feed. This situation makes the price for castor meal is lower compared to the prices of soy meal and rapeseed meal as both type of the meal can be used as animal feed. The high fertilizer value in the castor meal with the lower price in the market makes the demand for the castor meal increase from the organic fertilizer market.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 MATERIAL AND SOLVENT

Hexane (C_6H_{14}) is the solvent that will be used to extract the castor oil. The mixture of diethyl ether (C_2H_5)₂O and ethanol (C_2H_5OH) and few drop of phenolphthalein with the titration process using 0.1M NaOH is used to determine the acid value of oil. After that, 0.1N ethanolic potassium hydroxide with few drop of phenolphthalein will be used to obtain the saponification value of the oil. The carbon tetra chloride (CCl_4) will be added with Wijs solution and aqueous potassium iodide (KI) and then be titrating with 0.1M sodium-thiosulphate solution ($Na_2S_2O_3$) to determine the iodine value of the oil. The starch indicator is added when the process to determine the iodide value of the oil. HCl is used to active the clay in the refining process of extracted castor oil. NaOH and the sodium chloride are added in the neutralization process of the extracted oil. Concentrated sulphuric acid (H_2SO_4) is used in the modification process of the oil that obtains from the extraction process and then be neutralizing using sodium hydroxide (NaOH).

3.2 APPARATUS

The apparatus that will be used in this experiment are oven, soxhlet extractor and viscometer. Detail explanations are provided in Section 3.2.1, 3.2.2, and 3.2.3.

3.2.1 Oven

Oven as illustrated in Figure 3.1 is the equipment that will be used in this research for drying purposes. The oven is designed with maximum temperature of 220°C. The front panel located at the top of the unit provides a digital display of the temperature reading in Centigrade. It has three levels of tray and drying process can be set up by wall heat or by blowing hot air from the fan on the wall at the inside back of the oven. Time consumption of drying process can be set up on lower part of front panel so that the oven will be automatically reduce its temperature to ambient temperature after drying process end.



Figure 3.1: Laboratory oven

3.2.2 Soxhlet extractor

The soxhlet extraction is normally used when the desired product or compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. The simple filtration can be used to separate the compound from the insoluble substance if the desired compound has a significant solubility in a solvent. The material that containing some of the desired product will be placed inside the thimble that made from the thick filter paper which is placed into the main chamber of the soxhlet extractor. Then, the soxhlet extractor is placed onto a flask which containing the extraction solvent and equipped with a condenser. During the extraction process, the flask that contains the solvent is heated to reflux. The solvent will vaporize up to distillation arm, and then flood into the chamber housing the thimble of the material. The used of condenser is to make sure that the solvent vapor that already cools drips back down into the chamber housing the material. The chamber slowly fills with warm condensate solvent. Some of the desired compounds will the dissolve in the solvent. As the extractor is almost full, the chamber will automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask and the cycle will be allowed to repeat many times.



Figure 3.2: Laboratory soxhlet extractor

3.2.3 Viscometer

Viscometer which is also known as viscosimeter is one type of laboratory equipment which is used to measure the viscosity of a fluid. An instrument called a rheometer is used to measure the liquid with the viscosities that vary with flow condition. The viscometer is used to measure only one flow condition. In general, either the fluid remains stationary and an object moves through it, or the object is stationary and the fluid moves past it. The drag caused by relative motion of the fluid and a surface is a measure of the viscosity. The flow conditions must have a sufficiently small value of Reynolds number for there to be laminar flow.



Figure 3.3: Laboratory viscometer

3.3 EXPERIMENTAL WORK

The experimental work is shown in the figure 3.4. The experiment start with the determination of oil content and then the oil is characterize. After that, the extracted oil is refined and then be characterize again. Lastly, the refined oil is modified using sulfation method.

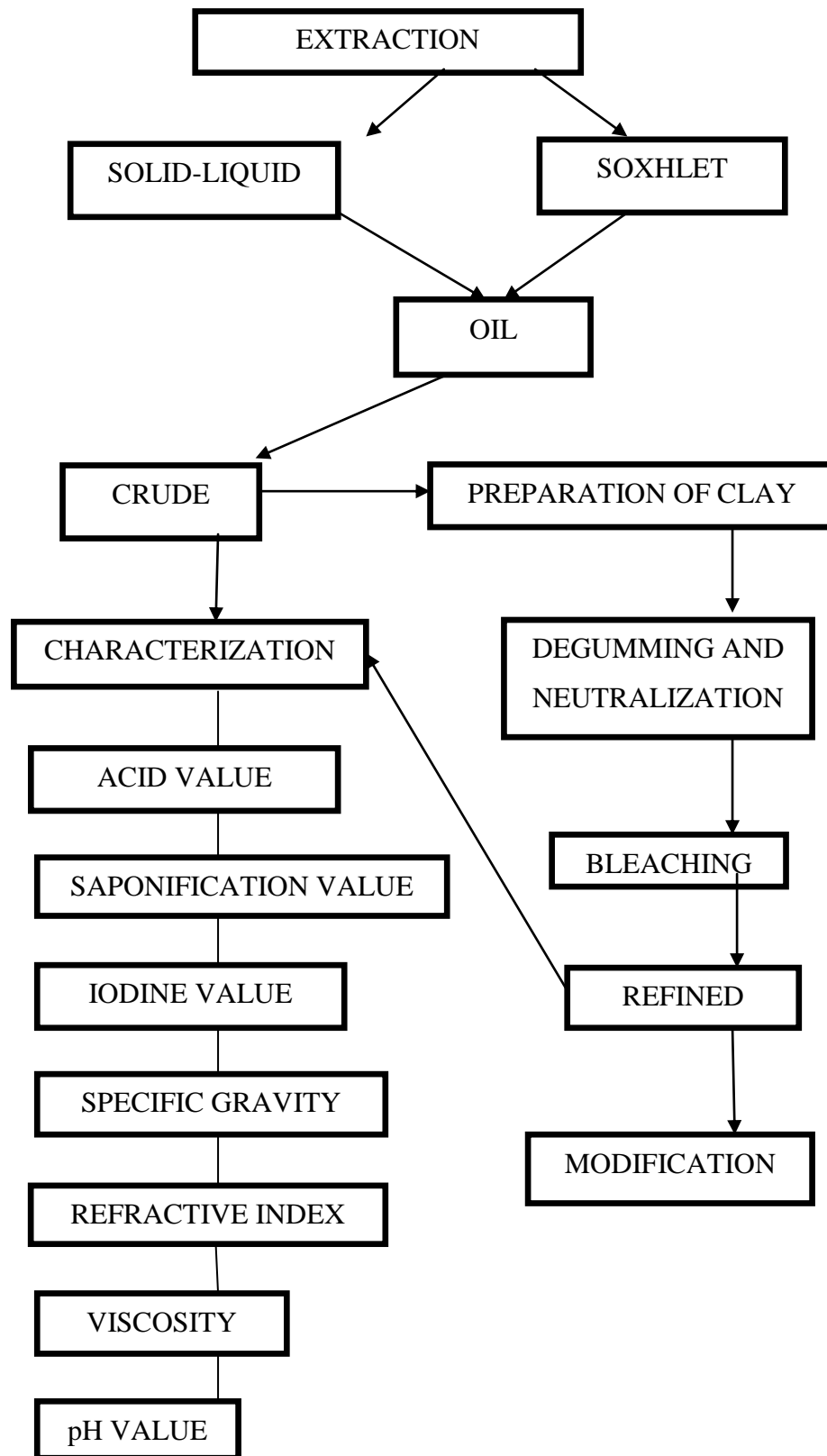


Figure 3.4: Flow of the experimental work

3.3.1 Castor Beans Processing

The castor beans must undergo some step of processing before the extraction procedure. Firstly, the castor seeds are cleaned as the seeds which are obtain from hand picking, had some foreign material and dirt. The cleaned castor seeds then placed in open area for sun dried until the casing splits and sheds the seeds. After sun dried, the beans then go for further dried as the beans are place inside the oven at 60°C for 7 hour to reduce its moisture content which normally castor bean contain about 5 to 7% of moisture. After that, the shell is separated from the nibs (cotyledon) by using the tray that blows away the cover in order to achieve high yield. By using the mortar and pestle, the castor beans are crush into a paste which is called ‘cake’ to weaken the cell wall to release castor fat during the extraction.

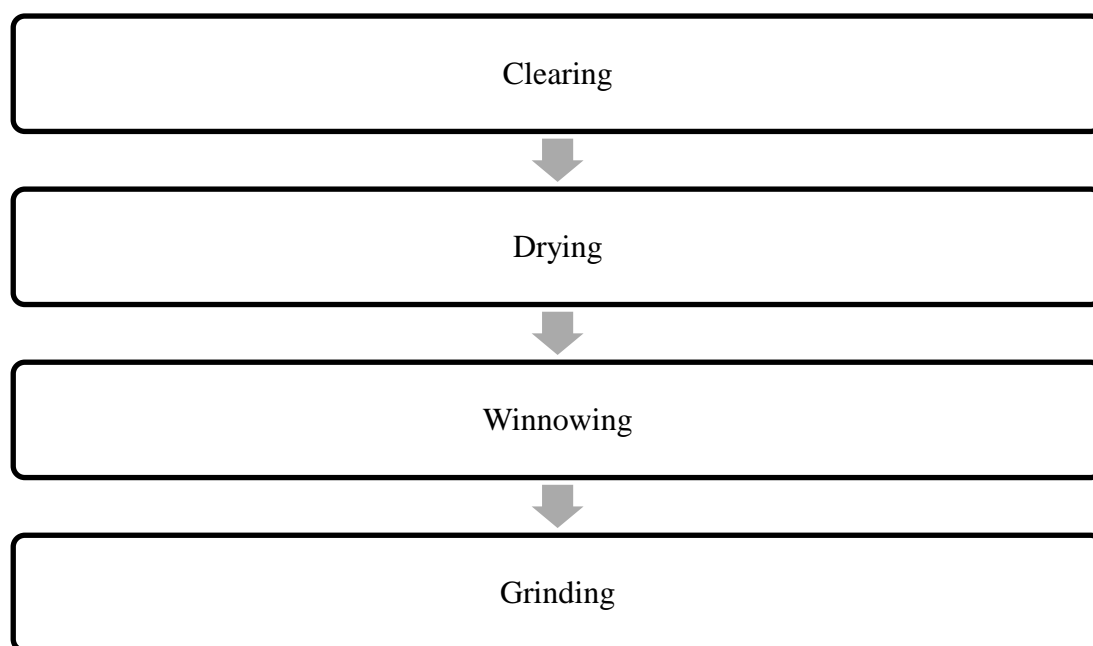


Figure 3.5: Flow chart of castor bean processing

3.3.2 Operation of Soxhlet Extractor

10g of the castor bean cake is placed in the thimble and inserted in the center of the soxhlet extractor. 300ml of normal hexane is poured into a round bottom flask. The soxhlet then heated at 60°C and as the solvent is boiling, the vapor rises until the condenser at the top of the extractor. The condensate solvent then drip into

the filter paper thimble in the center of the thimble which contain the castor cake to be extracted. The extracted product then seeps through the thimble's pores and flow back down into the round bottom flask. The extraction is proceeding until 30 minutes. After that, the castor cake is remove from the tube and dried in the oven and be cooled inside the desiccators. The castor cake is weighted again to determine the amount of the oil that already extracted. The further extraction is proceeding at 30 minutes interval until the cake weighed at further extraction and the previous extraction is same. The extraction then carried out for 5g of the castor cake. The weight of the oil extracted is determined at 30 minutes time interval. The final step to obtain the extracted castor oil is the mixture of the solvent and the extracted oil is heated to recover the solvent from the oil.

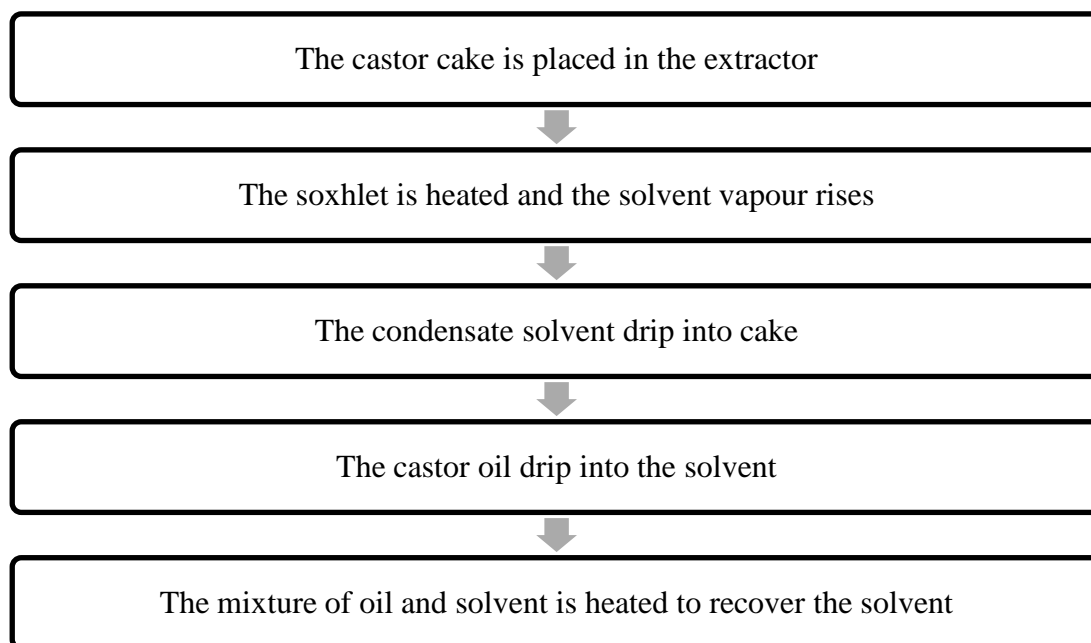


Figure 3.6: Flow chart of operation of soxhlet extractor

3.3.3 Determination of Moisture Content of the Seeds

The cleaned castor seeds are weighed and then dried inside an oven that operates at 80°C for 7 hours and the weight of the seed is take after at every 2 hours. The step is repeated until the weight of the seed is constant. During the 2 hours interval, the seeds are removed from the oven and the cooled inside the desiccators for 30 minutes. The cooled seeds then remove from the desiccators and re-weighed. The percentage of the moisture inside the castor seeds is calculated using the

formula: Moisture= $100(W_1 - W_2) / W_2$ %, where W_1 = original weight of the castor seed before drying and W_2 = weight of the sample after drying.

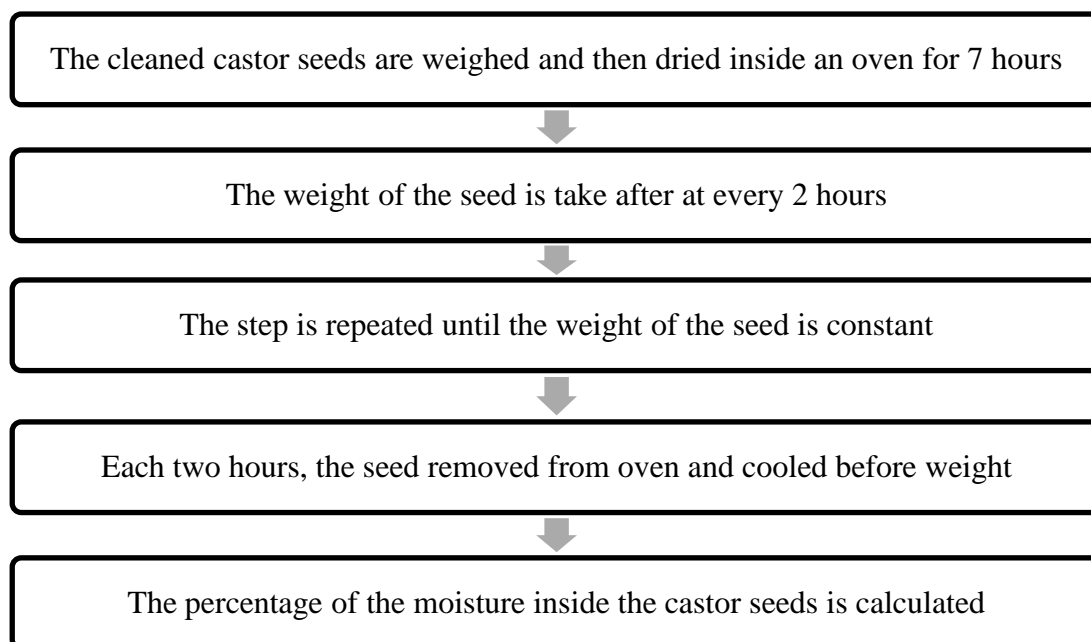


Figure 3.7: Flow chart of determining the moisture content

3.3.4 Determination of the Percentage of Castor Oil Extracted

30g of castor seed that already crushed into the castor cake is place inside the thimble and 300ml of the hexane as poured into the round bottom flask. The soxhlet is heated at 60°C and the extraction is allowed for 3 hours continuously extraction. The procedure is repeated by using the different weights of the castor cake. The final step is the solvent which is the hexane, is removed by distillation process and the percentage of extracted oil is determined.

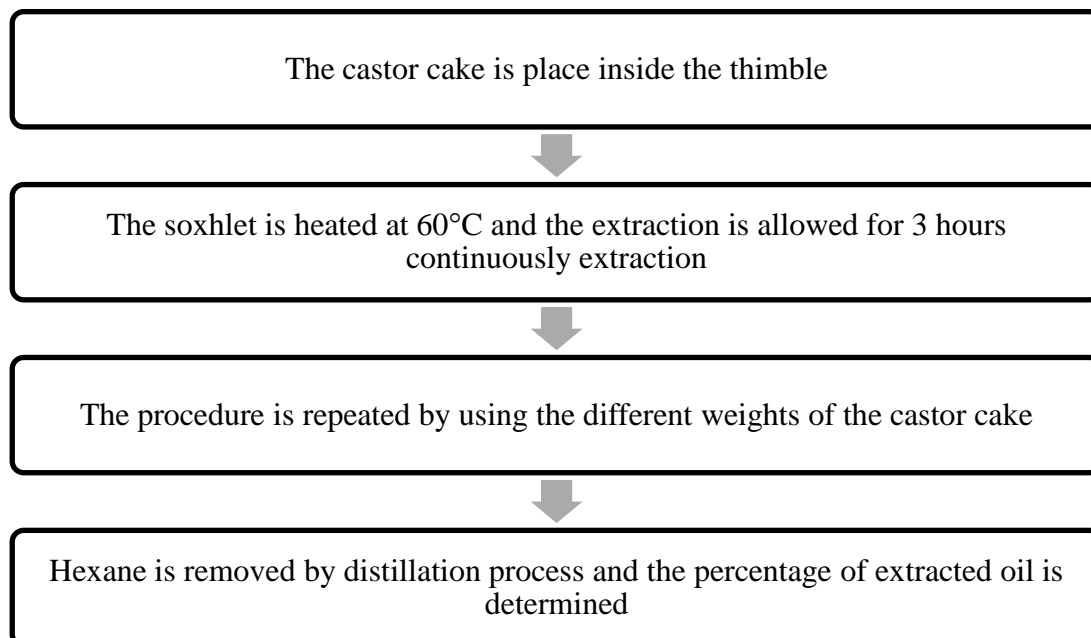


Figure 3.8: Flow chart of determining the percentage of oil extracted

3.3.5 Characterization of the Extracted Castor Oil

3.3.5.1 Determination of Acid Value

Firstly, 25ml of diethyl ether and 25ml of ethanol is mixed inside a 250ml beaker and the resulting mixture is added to 10g of extracted oil in 250ml conical flask. A few drops of the phenolphthalein are added inside the conical flask as the indicator. The mixture then titrated with 0.1M NaOH with consistent shaking until the titration is at the end point as a dark pink colour is observed. The volume of the 0.1M NaOH (V_0) that used in the titration process is noted. The free fatty acid (FFA) is calculated by using the formula: $V_0/W_0 \cdot 2.82 \cdot 100$, where 100ml of 0.1M NaOH = 2.83g of oleic acid, W_0 = sample weight, then acid value = $FFA \cdot 2$

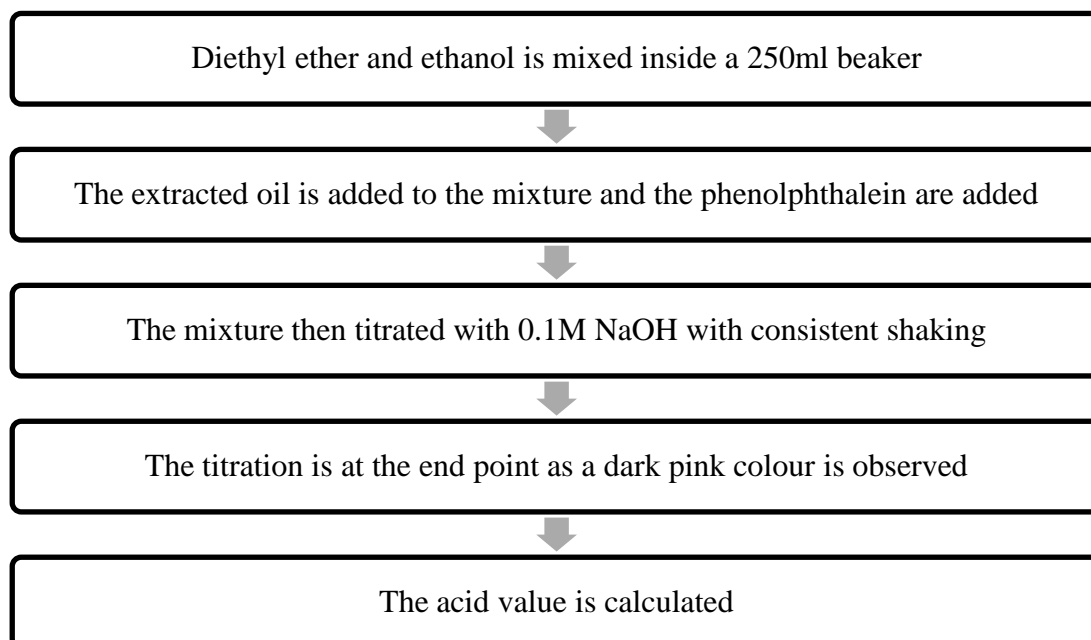


Figure 3.9: Flow chart of determining the acid value

3.3.5.2 Determination of Saponification Value

25ml of ethanolic potassium hydroxide is added to 2g of the extracted oil inside the conical flask. The mixture is consistently stirred and is boiled for 60 minutes. The reflux condenser is placed on the top of the flask. A few drops of the phenolphthalein are added to the warm solution and then is titrated by 0.5M HCl until the pink colour of the indicator is disappeared. The same procedures are used on the other sample and blank. The expression used to show the saponification value is given by the formula: $(S.V) = 56.1N (V_0 - V_1)/M$, where V_0 = the volume of the solution used for blank test, V_1 = the volume of the solution used for determination, N = Actual normality of the HCl used, M = Mass of the sample.

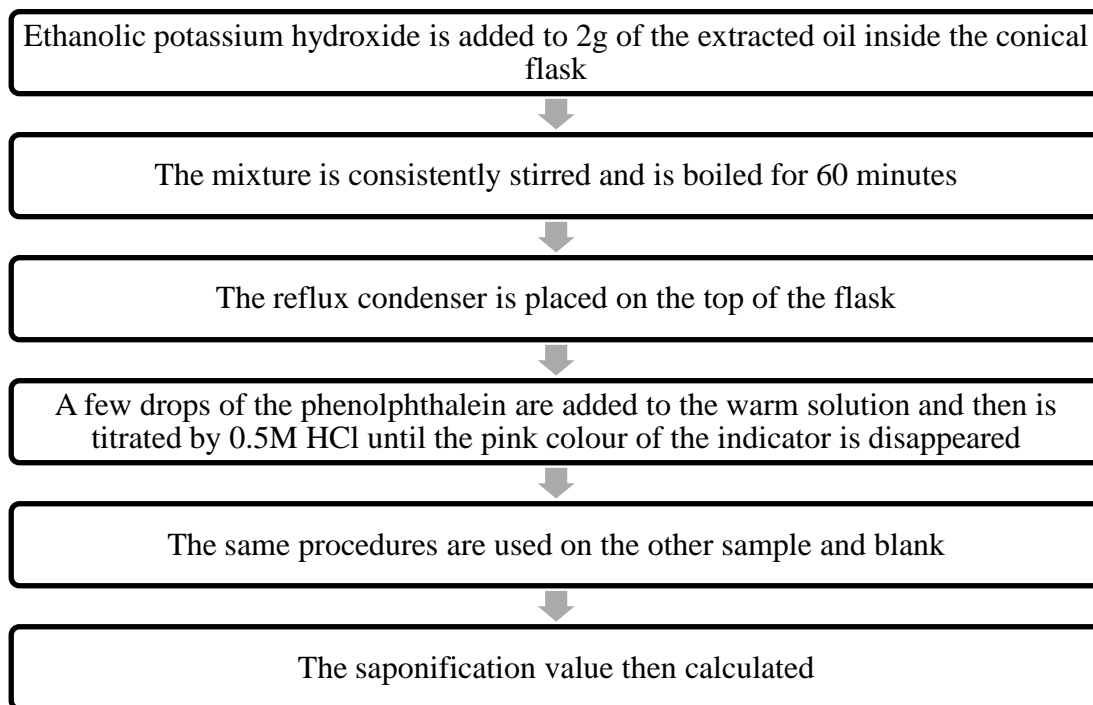


Figure 3.10: Flow chart of determining the saponification value

3.3.5.3 Determination of Iodine Value

20ml of carbon tetra chloride is added inside the conical flask to dissolve 0.4g of the extracted castor oil. After that, 25ml Wijs solution is added to the conical flask using a safety pipette in the fume chamber. The stopper is inserted on the top of the flask and the content of the flask is swirled vigorously. Then, the flask is placed in the dark place for 30 minutes. At the end of this period, 125ml of water and 20ml of 10% aqueous potassium iodide are added into the flask using the measuring cylinder. The content of the flask is titrated with 0.1M sodium-thiosulphate solution to end point until the yellow colour almost disappeared. 3ml of starch solution are added and the titration process is continued by adding thiosulphate drop until the blue colour disappeared after vigorous shaking. The procedures are used for other samples and blank test. The iodine is determined by the formula: $(0I.V) = 12.69C (V_1 - V_2)/M$, where C = Concentration of sodium thiosulphate used, V_1 = Volume of sodium thiosulphate used for blank, V_2 = Volume of sodium thiosulphate used for determination, M = Mass of the sample.

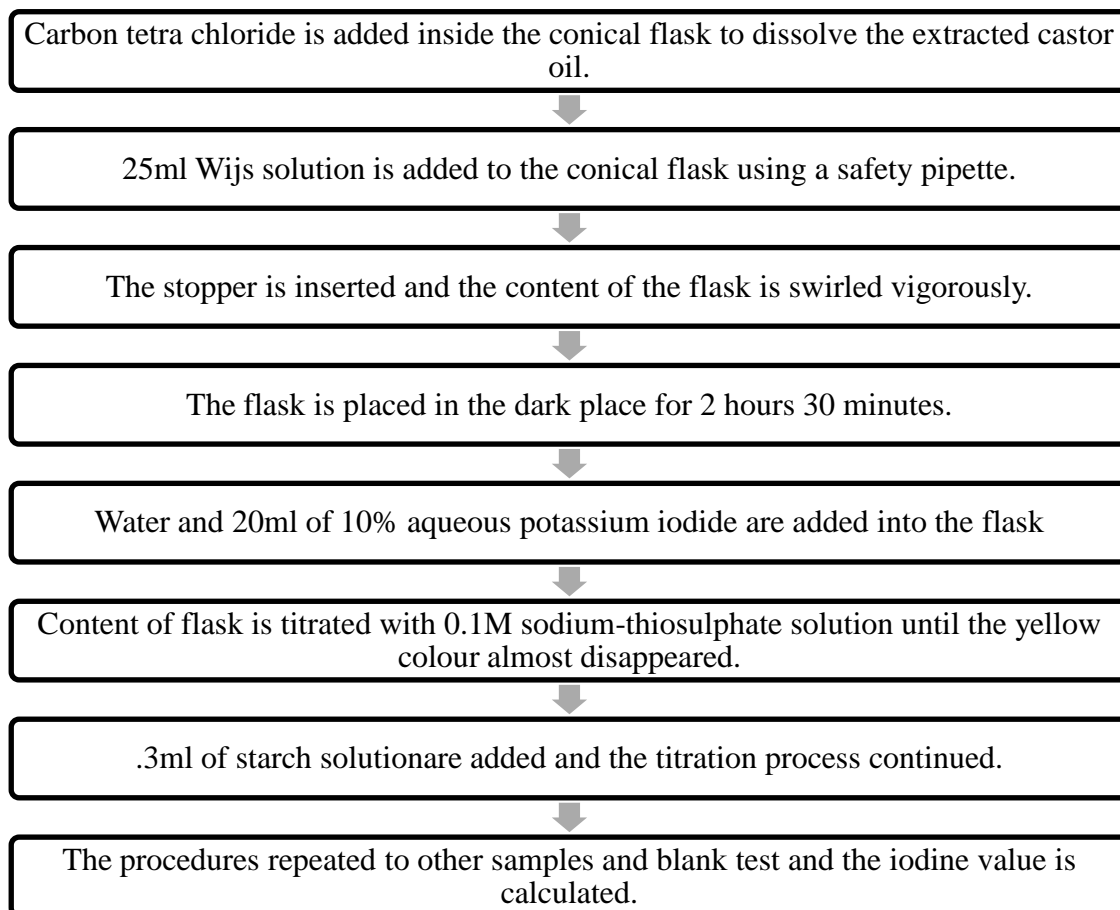


Figure 3.11: Flow chart of determining the iodine value

3.3.5.4 Determination of Specific Gravity

Density of the bottle is used to determine the density of the extracted oil. Firstly, a cleaned and dry bottle of 25ml is weighed (W_0) and the bottle then filled with the oil with the stopper is inserted. The bottle then re-weighed to give (W_1). After that, the oil is removed from the bottle and the bottle is washed and dried before the water is inserted into the bottle. The water with the water inside is weighed to give (W_2). The specific gravity is determined by using the formula:

$$(W_1 - W_0) / (W_2 - W_0) = \text{mass of the substance} / \text{mass of an equal volume of water.}$$

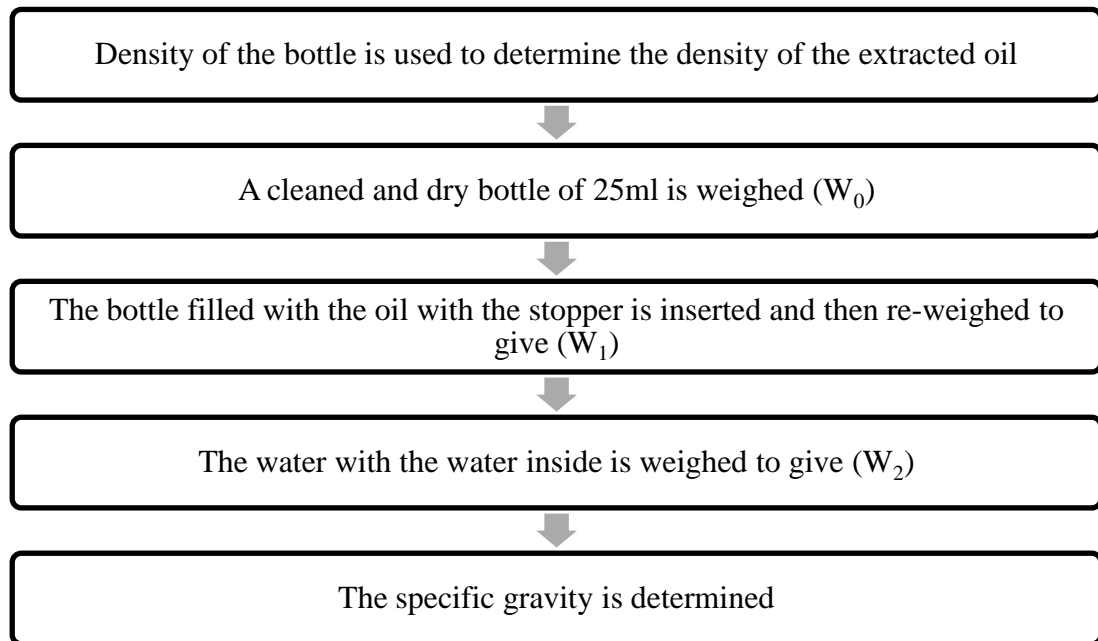


Figure 3.12: Flow chart of determining the specific gravity

3.3.5.5 Determination of Viscosity

The viscosity of the castor oil can be determined by using the viscometer that available in laboratory. The type of spindle used is S03 and the speed of spindle stir is at 10rpm in the room temperature which is 25°C . The water bath is set up to the room temperature and the rpm for the spindle is set up before the start the viscometer. The viscosity of the castor oil is showed at the viscometer screen panel.

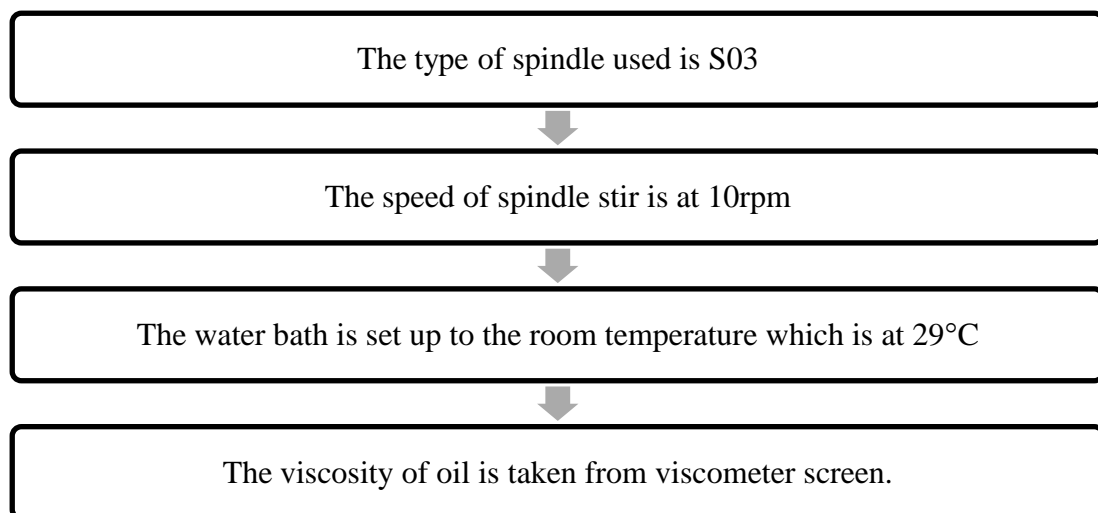


Figure 3.13: Flow chart of determining the viscosity of the castor oil

3.3.5.6 Determination of Refractive Index

The refractometer is used to determine the refractive index of the castor oil. A few drops of the castor oil are transferred into the glass slide of the refractometer. At no parallax error, the value of the refractive index is observed and recorded.

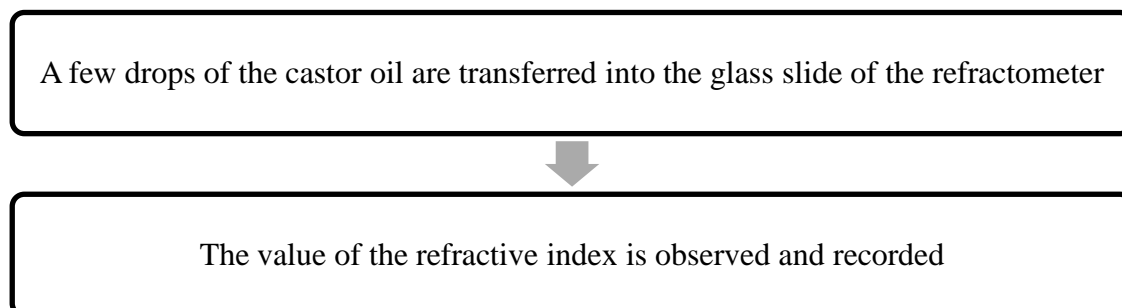


Figure 3.14: Flow chart of determining the refractive index

3.3.5.7 Determination of pH Value

2g of the oil sample is poured into a clean dry 25ml beaker. 13ml of hot distilled water is added to the oil sample in the beaker and then slowly stirred. The mixture is then cooled in a cold-water bath to 25°C. The pH electrode is standardized with the buffer solution and the electrode is immersed into the sample and the pH value is observed and recorded.

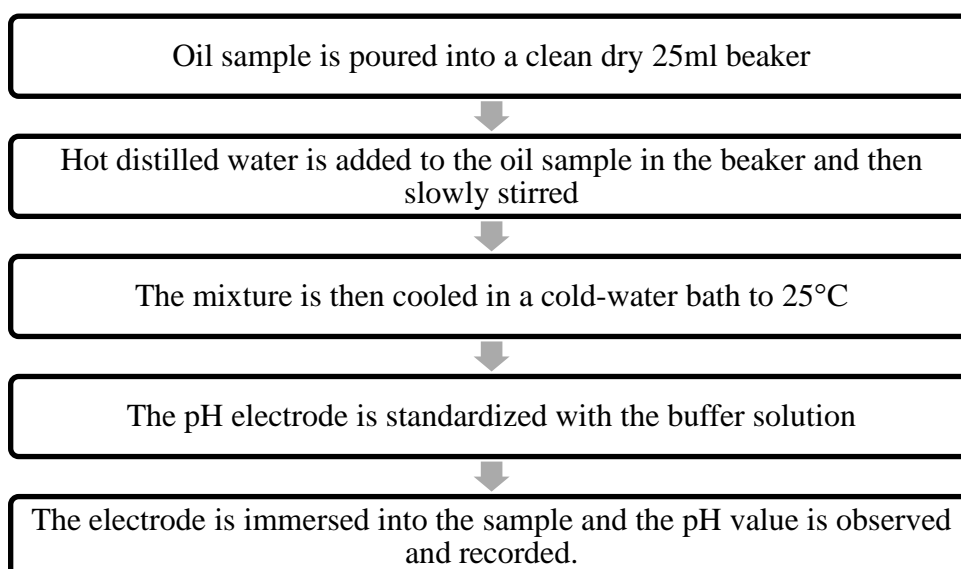


Figure 3.15: Flow chart of determining the pH value

3.3.6 Refining of Extracted Castor Oil

3.3.6.1 Preparation of Clay

The clay is obtained from the ground and mixed with the water. The impurities inside the clay such as sand and stone are removed. 2M HCl is added to the clay slurry and the mixture is boiled at 100°C for 2 hours. After that, the mixture is washed with water to remove the acid, dried and ground.

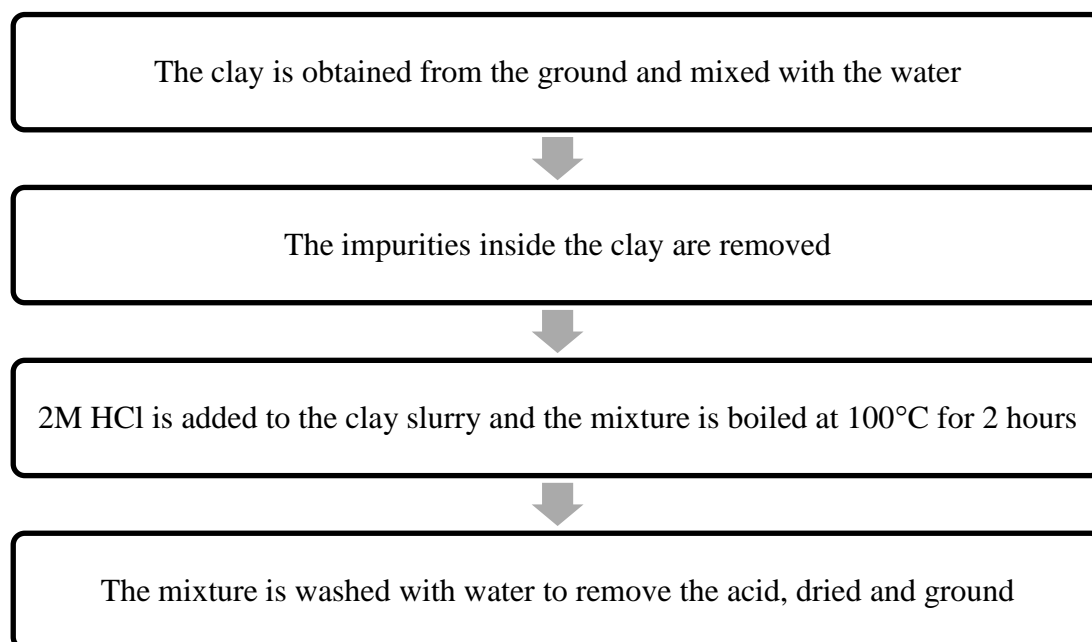


Figure 3.16: Flow chart of preparation of clay

3.3.6.2 Degumming and Neutralization

The extracted castor oil is degummed by adding the boiled water and the mixture is stirred for 2 minutes and then the mixture is allowed to stand in the separation funnel. Thereafter, the aqueous layer that exists is removed. The same procedure is repeated to make sure most gums is removed. The neutralization of the oil is started with the degummed oil is poured into the beaker and then heated to 80°C and then 40ml of 0.1M NaOH is added as to help settle out the soap formed. The mixture is transferred into the separating funnel and allowed to stand for 1 hour. The soap that formed is separated from the oil and the hot water is added again and

again to the oil solution until the soap remaining in the solution is removed. Lastly, the neutralize oil is drawn into a cleaned and dried beaker.

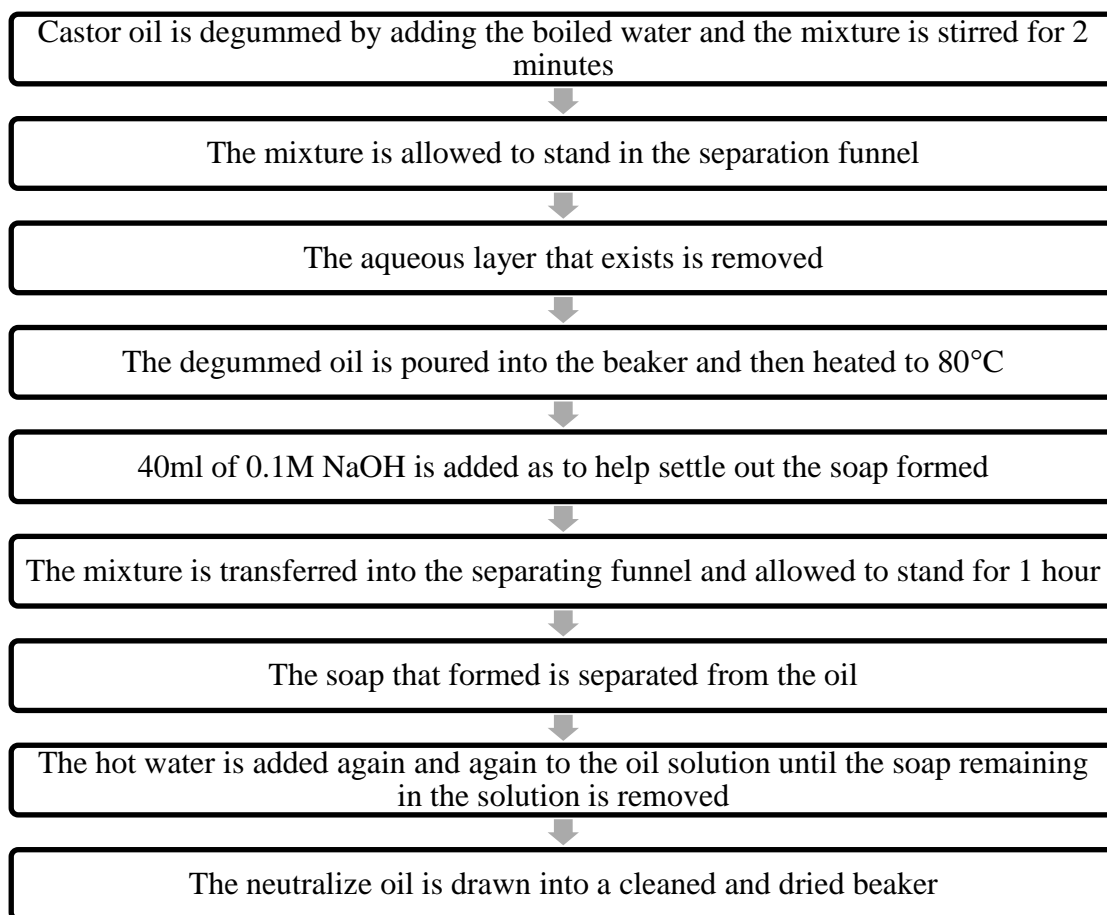


Figure 3.17: Flow chart of degumming and neutralization process

3.3.6.3 Bleaching

The neutralize oil is poured into a beaker and then the oil is heated to 90°C. The activated clay (15% by weight of oil) is added to the heated oil. The mixture is stirred for 30 minutes and the temperature is allowed to rise until 110°C for another 30 minutes. The product lastly filtered at 70°C.

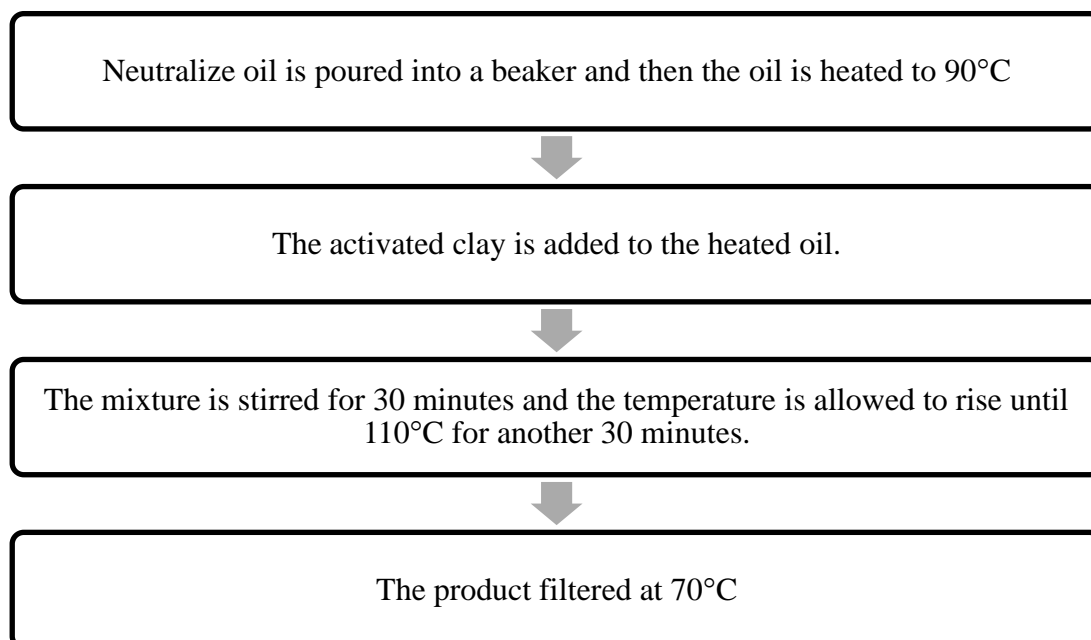


Figure 3.18: Flow chart of bleaching process

3.3.7 Modification of the Refined Castor Oil (Sulphation)

The refined castor oil warmed at 35°C and then added with 15ml of concentrated sulphuric acid (98%). The mixture is allowed to react with constant stirring. After that, the product is washed by using hot distilled water and then left to stand for about 2 hours. The water then removed and the sulphuric acid ester that formed is finally neutralized with 10ml of 0.1m sodium hydroxide.

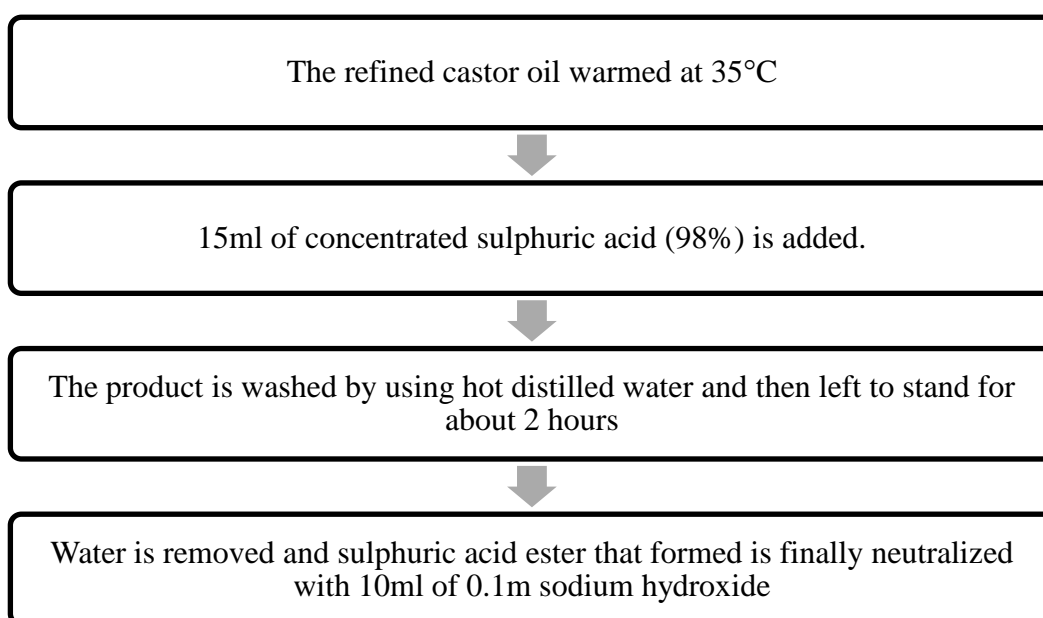


Figure 3.19: Flow chart of modification of refined castor oil

CHAPTER 4

RESULT AND DISCUSSION

4.1 QUALITY OF DIFFERENT CASTOR SEED

The castor seed nowadays is planted in many places in Malaysia. It is believed that the different locations the castor seed is planted gives different quality and quantity of oil. In this research, there are two types of castor seed that will be characterize. Both of the castor seed are labeled with the name Casa 5 and Casa 101. Casa 5 and Casa 101 comes from the different placed in Sabah and using the various formula in the experiment procedure, both types of the castor seed are characterize for the moisture content, physical properties, chemical properties and the percentage of oil extracted.

4.2 ASTM VALUE FOR CASTOR OIL

The castor oil that extracted from Malaysian castor seeds were compared to the world castor oil quality. The American Society for Testing and Material (ASTM) specification will be used as ASTM is an international standard organization that develops and also publishes voluntary consensus technical standard for a wide range of materials, products, systems and services. So the Malaysian castor oil were compared to ASTM specification for quality castor oil as shown in table 4.1.

Table 4.1: ASTM specification for Quality Castor Oil

Property	Range
Specific gravity 20/25 °C	0.957-0.968
Refractive Index, nD	1.476-1.479

Saponification Value	175-187
Un-saponification Value	0.3-0.7
Iodine value	82-88
Hydroxyl Value	160-168
Viscosity at 25°C	6.3-8.8 St
Acid Value	0.4-4.0
Colour	Not darker than 2-3

4.3 CASA 5

Table 4.2: Determination of Moisture Content

Time (g)	0	2	4	6	7
Weight (g)	18.0	17.84	17.57	17.24	17.23

Moisture content = 4.28%

Table 4.3: Physical Properties of the Crude and Refined Castor Oil

Property	Crude Castor Oil	Refined Castor Oil
Specific gravity	0.9578	0.9578
Viscosity	7.33	6.38
Refractive Index	1.4752	1.4773
pH	6.4	7.27
Colour	Amber	Amber

Table 4.4: Chemical Properties of the Crude and Refined Castor Oil

Property	Crude Castor Oil	Refined Castor Oil
Acid Value	3.3	3.2
Saponification Value	178.12	176.72
Iodine Value	86.61	84.39

Table 4.5: Determination of Percentage of Castor Oil Extracted

Determination	Value (g)
Weight of seed	47.1
Weight of kernel	36.2
Weight of husk	10.8
Weight of oil	14.8

Percentage of oil extracted=40.88%

4.3.1 Discussion(Casa 5)

The percentage value for the moisture content for the Casa 5 is 4.28%. This shows that the value are falls in the reported range of the moisture content which is 5 to 7 percent. The result obtained for the percentage of oil extracted using Soxhlet Extraction for Casa 5 is 40.88% which falls within the range of the oil content (30 to 55%) of castor seeds. The oil percentage value can increase if the type of the extraction used is by the use of hydraulic pressing followed by solvent-extraction process. The table 4.3 present a comparison in the physical properties between crude oil and refined oil obtained in the experiment for Casa 5. The specific gravity for both crude and refined oil obtained have the same value; 0.9578. This value is still in ASTM range. The viscosity for the crude and refined oil shows the different value. The crude oil viscosity is 7.33st and the value of the viscosity for refined oil is 6.38st which both of the value still fall in the ASTM range. The viscosity value for refined is more lower than the crude oil because this may happened as some impurities and some other components were removed during the refining process. The result obtained for the refractive index for both crude and refined oil is 1.4752 and 1.4773. This little different can be acceptable as both of the value still in ASTM specification.

Moreover, the pH value for the crude and refined oil is 6.4 and 7.27 respectively. This shows that the crude oil is more acidic compared to the refined oil which may be result from the neutralization and degumming process during the

refining process. The chemical properties analysis for Casa 5 shows in table 4.4. The acid value for crude oil is 3.3 mg NaOH/ g of oil and for refined oil is 3.2 mg NaOH/ g of oil. The small different in this two value may happened as some fatty acid is neutralized in refining process. However, both values still fall in the ASTM standard. Besides that, the saponification value for the crude and refined oil found to be 178.12 mg KOH/ g of oil and 176.72 mg KOH/ g of oil. This data shows that the value for the crude is higher than refined as more alkaline is needed to neutralize the available fatty acid in the crude oil compared to the refined oil. Both of the values is compared to the ASTM specification and the value is still in the range of ASTM standard. The result obtained for the iodine value for crude and refined oil is 86.61 and 84.39. This data shows that the amount of iodine that capable to absorbed by unsaturated acid are more higher for the crude oil compared to the refined oil. The iodine values that obtain from the experiment is compared with the ASTM standard shows in Table 4.1. Both of the virgin(crude) and refined oil can be classified as a non-drying oils as the iodine values for both of the oil are lower than 100. So, the castor oil could be use as lubricant and also hydraulic brake fluids.

4.4 CASA 101

Table 4.6: Determination of Moisture Content

Time (g)	0	2	4	6	7
Weight (g)	71.8	70.2	69.41	68.85	68.83

Moisture content = 4.14%

Table 4.7: Physical Properties of the Crude and Refined Castor Oil

Property	Crude Castor Oil	Refined Castor Oil
Specific gravity	0.9579	0.9578
Viscosity	7.44	6.576
Refractive Index	1.4745	1.4784
pH	6.7	7.2
Colour	Amber	Amber

Table 4.8: Chemical Properties of the Crude and Refined Castor Oil

Property	Crude Castor Oil	Refined Castor Oil
Acid Value	2.3	1.8
Saponification Value	180.92	175.31
Iodine Value	85.02	82.17

Table 4.9: Determination of Percentage of Castor Oil Extracted

Determination	Value (g)
Weight of seed	92.7
Weight of kernel	71.8
Weight of husk	20.5
Weight of oil	26.6

Percentage of oil extracted=37.04%

4.4.1 Discussion(Casa 101)

The percentage value for the moisture content for the Casa 101 is 4.14%. This shows that the value are falls in the reported range of the moisture content which is 5 to 7 percent. The result obtained for the percentage of oil extracted using Soxhlet Extraction for Casa 101 is 37.04% which falls within the range of the oil content (30 to 55%) of castor seeds. The oil percentage value can increase if the type of the extraction used is by the use of hydraulic pressing followed by solvent-extraction process. The table 4.7 present a comparison in the physical properties between crude oil and refined oil obtained in the experiment for Casa 101. The specific gravity for both crude and refined oil obtained have the very small different value which is 0.9579 for crude oil and 0.9578 for refined oil. This value is still in ASTM range. The viscosity for the crude and refined oil shows the different value. The crude oil viscosity is 7.44st and the value of the viscosity for refined oil is 6.576st which both of the value still fall in the ASTM range. The viscosity value for refined is more lower than the crude oil because this may happened as some impurities and some other components were removed during the refining process.

The result obtained for the refractive index for both crude and refined oil is 1.4745 and 1.4784. This little different can be acceptable as both of the value still in ASTM specification.

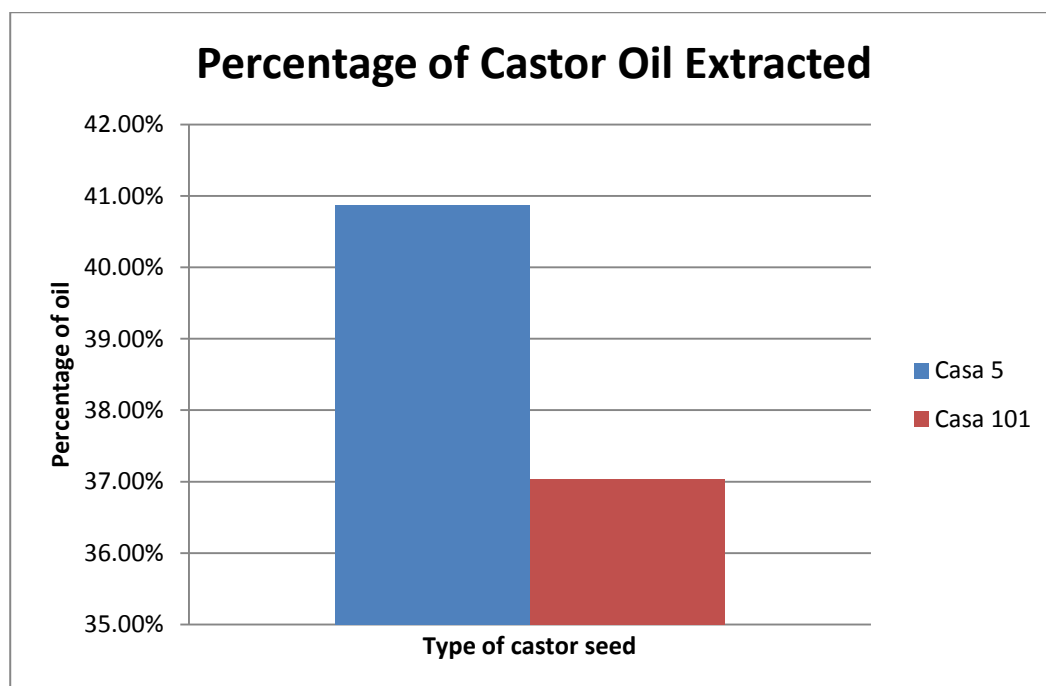
Moreover, the pH value for the crude and refined oil is 6.7 and 7.2. This shows that the crude oil is more acidic compared to the refined oil which may be result from the neutralization and degumming process during the refining process. The chemical properties analysis for Casa 101 shows in table 4.8. The acid value for crude oil is 2.3 mg NaOH/ g of oil and for refined oil is 1.8 mg NaOH/ g of oil. The small different in this two value may happened as some fatty acid is neutralized in refining process. However, both values still fall in the ASTM standard. Besides that, the saponification value for the crude and refined oil found to be 180.92 mg KOH/ g of oil and 175.31 mg KOH/ g of oil. This data shows that the value for the crude is higher than refined as more alkaline is needed to neutralize the available fatty acid in the crude oil compared to the refined oil. Both of the values is compared to the ASTM specification and the value is still in the range of ASTM standard. The result obtained for the iodine value for crude and refined oil is 85.02 and 82.17. This data shows that the amount of iodine that capable to absorbed by unsaturated acid are more higher for the crude oil compared to the refined oil. The iodine values that obtain from the experiment is compared with the ASTM standard shows in Table 4.1. Both of the virgin(crude) and refined oil can be classified as a non-drying oils as the iodine values for both of the oil are lower than 100. So, the castor oil could be use as lubricant and also hydraulic brake fluids.

4.5 COMPARISON CASA 5 AND CASA 101 PERCENTAGE OF OIL AND QUALITY WITH ASTM STANDARD

The experimental result that obtain from the calculation for the physical properties and chemical properties of the Casa 5 and Casa 101 castor oil is compared with the ASTM standard. The comparison is showed in the table 4.10. From the observation that be made for both of the Casa 5 and Casa 101, the quality of castor oil for Casa 5 and Casa 101 is same as both of extracted oil followed the ASTM standard. The percentage of oil obtained from Casa 5 and Casa 101 is compared and showed in figure 4.1

Table 4.10: Comparison Casa 5 and Casa 101 Quality with ASTM Standard

Property	Range (ASTM)	Casa 5		Casa 101	
		Crude Castor Oil	Refined Castor Oil	Crude Castor Oil	Refined Castor Oil
Specific gravity	0.957-0.968	0.9578	0.9578	0.9579	0.9578
Viscosity	6.3-8.8 St	7.33	6.38	7.44	6.576
Refractive Index	1.476-1.479	1.4752	1.4773	1.4745	1.4784
pH	-	6.4	7.27	6.7	7.2
Colour	Not darker than 2-3	Amber	Amber	Amber	Amber
Acid Value	0.4-4.0	3.3	3.2	2.3	1.8
Saponification Value	175-187	178.12	176.72	180.92	175.31
Iodine Value	82-88	86.61	84.39	85.02	82.17

**Figure 4.1:** Comparison Casa 5 and Casa 101 percentage of oil

From graph showed in figure 4.1, the percentage of oil obtained from Casa 5 is more higher compared to Casa 101. This different percentage happened as Casa 5

and Casa 101 is planted at the different location. This showed that the ground or soil used to plant Casa 5 have the suitable condition to produce good castor seed that contained high percentage of oil.

4.6 PERCENTAGE OF CASTOR OIL EXTRACTED USING SOXHLET EXTRACTION VERSUS SOLID-LIQUID EXTRACTION

After the Malaysian Castor Seed is extracted to obtain the percentage of oil and physical and chemical properties by using the Soxhlet extractor, the castor seed is then be extracted using Solid-Liquid extraction process. This experiment is done to identified which type from this two extraction will give more oil percentage. The type of castor seed used in this determination is Casa 5. The comparison of the percentage of oil obtained from Soxhlet extraction and Solid-Liquid extraction is showed in the table 4.11 and in figure 4.2.

Table 4.11: Determination of Percentage of Castor Oil Extracted using Soxhlet Extraction versus Solid-Liquid Extraction

Determination	Soxhlet Extraction	Solid-Liquid Extraction
	Value	Value
Weight of seed (g)	47.1	23.5
Weight of kernel (g)	36.2	18.0
Weight of husk (g)	10.8	5.4
Weight of oil (g)	14.8	7.8
Percentage of oil extracted	40.88%	43.3%

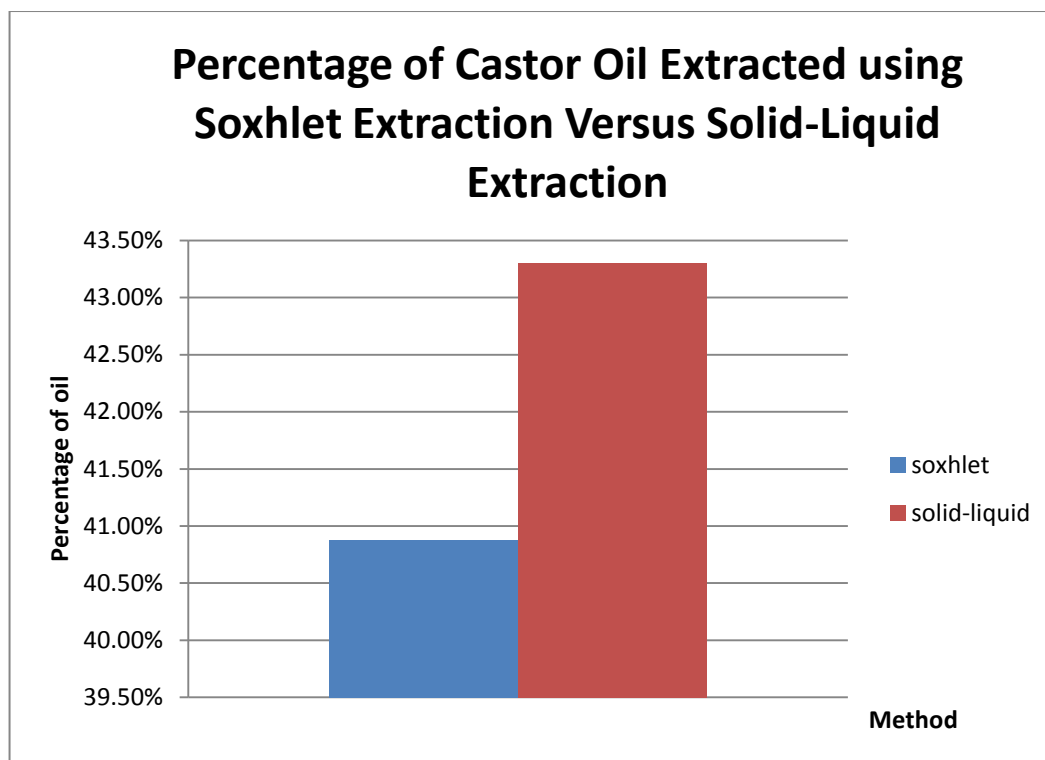


Figure 4.2: Comparison of oil obtained from Soxhlet Extraction and Solid-Liquid Extraction

From the table 4.11 and the graph showed in figure 4.2, the percentage of oil obtained from Solid-Liquid extraction is more higher compared to Soxhlet extraction. The explanation that can made is while using the Soxhlet extraction process, the crushed castor kernel is placed in the thimble and the condensed hexane is dripped to the thimble that contained the castor kernel, compared to the Solid-Liquid extraction process that the boiled hexane is directly contact with the crushed castor kernel. So, during Solid-Liquid extraction, hexane is more directly react with castor kernel to extract out the oil.

4.7 PERCENTAGE OF CASTOR OIL EXTRACTED USING CRUSHED CASTOR SEED VERSUS DEHULED CASTOR SEED

From the previous step, the castor seed is faced the dehul process which the castor kernel is separate from the castor husk and then, the castor kernel is extract to obtain the castor oil. This is done as it is believed that the castor husk did not contain the oil and only the castor kernel contained the oil. So, this experiment is done to extract the crushed castor seed which is contain castor kernel and husk and calculate

the percentage of oil obtained. The result of the percentage of oil is showed in table 4.12 and in figure 4.3. The process used is Soxhlet extraction process and castor seed used is Casa 5.

Table 4.12: Determination of Percentage of Castor Oil Extracted using Crushed Castor Seed Versus Dehuled Castor Seed

Determination	Dehuled Castor Seed	Crushed Castor Seed
	Value	Value
Weight of seed (g)	47.1	50.0
Weight of kernel (g)	36.2	-
Weight of husk (g)	10.8	-
Weight of oil (g)	14.8	27.0
Percentage of oil extracted	40.88%	54.0%

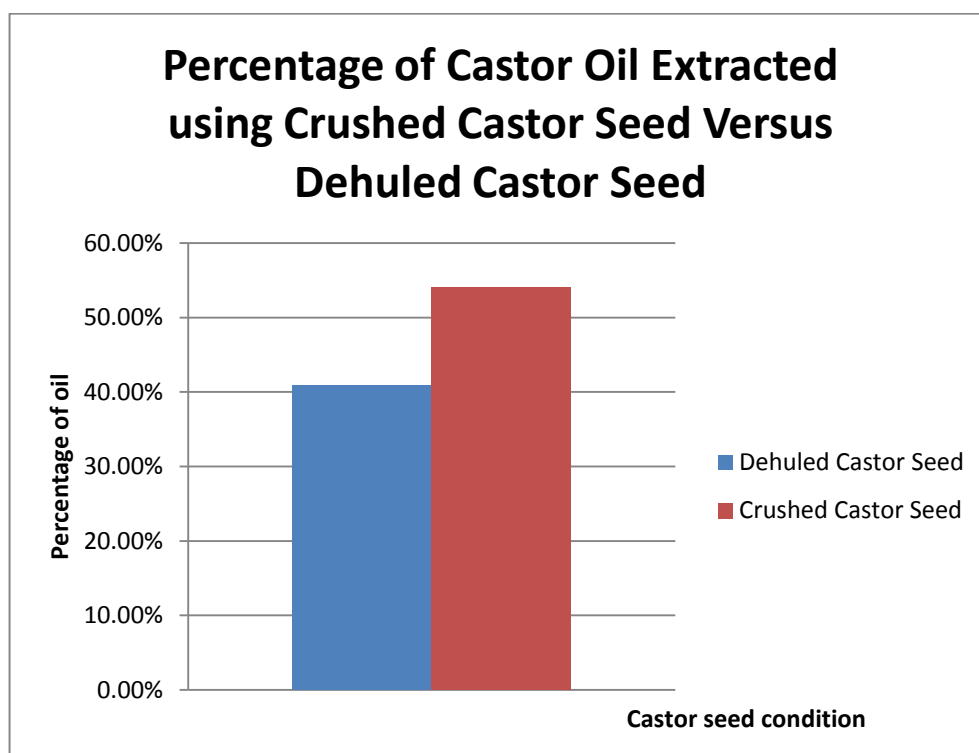


Figure 4.3: Comparison of oil obtained from dehuled castor seed and crushed castor seed.

From the result tabulated in table 4.12 and from graph showed in figure 4.3, the percentage of oil obtained from the crushed castor seed is more higher from

dehuled castor seed. This situation can be explain that as the dehul process is the process to separate the castor kernel from the husk. The castor kernel is extracted and the percentage of oil obtained is lower compared to the crushed seed. This showed that, there are some oil that left at the castor husk. This is the reason of why the crushed castor seed can achieved more higher percentage of oil compared to the dehuled castor seed. Although the crushed castor seed can give more percentage of oil, but from the observation made during this experiment, the extracted oil obtained has the dark brown color compared to the dehuled castor seed oil. This dark brown color comes from the castor husk in the crushed castor seed. This color can be reduced in the bleaching process.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The percentage of the castor oil obtained from Casa 5 is 40.88% of the total weight used compared to Casa 101 which obtained 37.04% of oil of the total weight used. The comparison is shown in the graph in figure 4.1. From the aspect of quantity of oil, this can conclude that Casa 5 can give more percentage of oil compared to Casa 101. Besides that, the castor oil extracted from the castor seed Casa 5 and Casa 101 is characterize and the value for physical and chemical properties is compared to the ASTM value. The comparison is showed in table 4.10. From the comparison of the value obtained from various experiment procedures with the ASTM standard, the quality of the castor oil for Casa 5 and Casa 101 have the same standard with the world standard specification of castor oil. The percentage of oil obtain from the Soxhlet extraction process is compared with the Solid-Liquid extraction process. The result showed in figure 4.2. The Solid-Liquid extraction gives more higher percentage oil compared to Soxhlet extraction. The castor seed is further extracted using the crushed castor seed and compared with dehuled castor seed. The graph obtained in figure 4.3 showed that the crushed seed gives more higher percentage of oil compared to dehuled seed. As this is the first characterization process made for Malaysian castor oil, this research give the new and the first information for the physical and chemical properties of the castor oil in Malaysia. So, the Malaysian castor seed can produced about high quantity of castor oil and have the same quality with world castor oil quality.

5.2 RECOMMENDATION

During the experiment take place in the laboratory, there were some ideas and recommendations derived from the research to build on a better future research.

The recommendations are listed as below:

- a) During the determination of the percentage of oil of Casa 5 and Casa 101, hexane is used as the solvent in the extraction process. It is recommended to use the different type of solvent to extract the castor oil from the castor seed. The other solvent that can be used like heptane or petroleum ether.
- b) In the determination of the percentage of oil of Malaysian castor seed, the method used is Soxhlet extraction and Solid-Liquid extraction. The other extraction process also can be used to determine the percentage of oil like microwave extraction process.

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APPENDIX A**CALCULATION OF MOISTURE CONTENT AND PERCENTAGE OF OIL
EXTRACTED FROM CASTOR SEED****CALCULATION****MOISTURE CONTENT**Casa 5

$$\begin{aligned}\text{Moisture content} &= \frac{18-17.23}{18} \times 100 \\ &= 4.28 \%\end{aligned}$$

Casa 101

$$\begin{aligned}\text{Moisture content} &= \frac{71.8-68.83}{71.8} \times 100 \\ &= 4.14\%\end{aligned}$$

PERCENTAGE OF OIL EXTRACTED

$$= \text{Kernel weight (g)} / \text{Oil obtain (g)} \times 100$$

Casa 5

$$\text{Kernel weight} = 36.2 \text{ g}$$

$$\text{Weight of extracted oil} = 14.8 \text{ g}$$

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{14.8}{36.2} \times 100 \\ &= 40.88\%\end{aligned}$$

Casa 101

$$\text{Kernel weight} = 71.8 \text{ g}$$

$$\text{Weight of extracted oil} = 26.6 \text{ g}$$

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{26.6}{71.8} \times 100 \\ &= 37.04\%\end{aligned}$$

APPENDIX B

CALCULATION OF PHYSICAL AND CHEMICAL PROPERTIES OF CRUDE CASTOR OIL

CALCULATION

ACID VALUE

V_0 = Titration Volume, mL

W_0 = Sample weight, 10g

FFA = $V_0 / W_0 \cdot 2.82 \cdot 100$

A.V = FFA .2

Casa 5

$V_0 = 2.2$ mL

$$V_0 = 2.2 \text{ mL} \times \frac{1L}{1000\text{mL}} \times \frac{2.13g}{\text{cm}^3} \times \frac{1 \text{ cm}^3}{0.001\text{dm}^3} \times \frac{1 \text{ dm}^3}{1L} \times \frac{1000 \text{ mg}}{1 \text{ g}}$$

$$= 4686 \text{ mg}$$

$$\text{FFA} = \frac{4686 \text{ mg}}{10 \times 2.82 \times 100}$$

$$= 1.6617$$

A.V = FFA x 2

$$= 1.6617 \times 2$$

$$= 3.3$$

Casa 101

$V_0 = 1.5$ mL

$$V_0 = 1.5 \text{ mL} \times \frac{1L}{1000\text{mL}} \times \frac{2.13g}{\text{cm}^3} \times \frac{1 \text{ cm}^3}{0.001\text{dm}^3} \times \frac{1 \text{ dm}^3}{1L} \times \frac{1000 \text{ mg}}{1 \text{ g}}$$

$$= 3195 \text{ mg}$$

$$\text{FFA} = \frac{3195 \text{ mg}}{10 \times 2.82 \times 100}$$

$$= 1.133$$

A.V = FFA x 2

$$= 1.133 \times 2$$

$$= 2.3$$

SAPONIFICATION VALUE

V_0 = Volume for blank test, 25.1 mL

V_1 = Volume for determination

Sample weight = 2 g

$$S.V = \frac{\text{Molecular weight for KOH} \times M \text{ of HCL used} \times (V_0 - V_1)}{\text{Sample weight}}$$

Casa 5

V_1 = 12.4 mL

$$S.V = \frac{56.1 \frac{g}{mol} \times 0.5 \frac{mol}{dm^3} \times (25.1 - 12.4) mL \times 1 \frac{dm^3}{1000 mL} \times 1000 \frac{mg}{g}}{2g}$$

$$= 178.12$$

Casa 101

V_1 = 12.2 mL

$$S.V = \frac{56.1 \frac{g}{mol} \times 0.5 \frac{mol}{dm^3} \times (25.1 - 12.2) mL \times 1 \frac{dm^3}{1000 mL} \times 1000 \frac{mg}{g}}{2g}$$

$$= 180.92$$

IODINE VALUE

V_0 = Volume for blank test, 47.1 mL

V_1 = Volume for determination

W = Sample weight, 0.4 g

C = Concentration of Sodium Thiosulphate used, 0.1M

$$I.V = \frac{126.9 \times C \times (V_0 - V_1)}{10 \times W}$$

Casa 5

$V_1 = 19.8$ mL

$$\begin{aligned} I.V &= \frac{126.9 \times 0.1 \times (47.1 - 19.8)}{10 \times 0.4} \\ &= 86.61 \end{aligned}$$

Casa 101

$V_1 = 20.3$ mL

$$\begin{aligned} I.V &= \frac{126.9 \times 0.1 \times (47.1 - 20.3)}{10 \times 0.4} \\ &= 85.02 \end{aligned}$$

SPECIFIC GRAVITY

W_0 = empty bottle weight, 9.5861 g

W_1 = Weight of bottle with oil

W_2 = Weight of bottle with water, 17.6563 g

$$\text{S.G} = \frac{(W_1 - W_0)}{(W_2 - W_0)}$$

Casa 5

W_1 = 17.3160 g

$$\begin{aligned} \text{S.G} &= \frac{(17.3160 - 9.5861)}{(17.6563 - 9.5861)} \\ &= 0.9578 \end{aligned}$$

Casa 101

W_1 = 17.3168 g

$$\begin{aligned} \text{S.G} &= \frac{(17.3168 - 9.5861)}{(17.6563 - 9.5861)} \\ &= 0.9579 \end{aligned}$$

VISCOSITY

Use spindle S03, at 10 rpm in the room temperature, 25°C

Casa 5

732.9cP = 7.33st

Casa 101

743.9cP = 7.44st

REFRACTIVE INDEX

Casa 5 = 1.4752

Casa 101 = 1.4745

pH VALUE

Casa 5 = 6.4

Casa 101 = 6.7

APPENDIX C

CALCULATION OF PHYSICAL AND CHEMICAL PROPERTIES OF REFINED CASTOR OIL

CALCULATION

ACID VALUE

V_0 = Titration Volume, mL

W_0 = Sample weight, 10g

FFA = $V_0 / W_0 \cdot 2.82 \cdot 100$

A.V = FFA .2

Casa 5

$V_0 = 2.1$ mL

$$V_0 = 2.1 \text{ mL} \times \frac{1\text{L}}{1000\text{mL}} \times \frac{2.13\text{g}}{\text{cm}^3} \times \frac{1 \text{ cm}^3}{0.001\text{dm}^3} \times \frac{1 \text{ dm}^3}{1\text{L}} \times \frac{1000 \text{ mg}}{1 \text{ g}}$$

$$= 4473 \text{ mg}$$

$$\text{FFA} = \frac{4473 \text{ mg}}{10 \times 2.82 \times 100}$$

$$= 1.586$$

$$\text{A.V} = \text{FFA} \times 2$$

$$= 1.586 \times 2$$

$$= 3.2$$

Casa 101

$V_0 = 1.2$ mL

$$V_0 = 1.2 \text{ mL} \times \frac{1\text{L}}{1000\text{mL}} \times \frac{2.13\text{g}}{\text{cm}^3} \times \frac{1 \text{ cm}^3}{0.001\text{dm}^3} \times \frac{1 \text{ dm}^3}{1\text{L}} \times \frac{1000 \text{ mg}}{1 \text{ g}}$$

$$= 2556 \text{ mg}$$

$$\text{FFA} = \frac{2556 \text{ mg}}{10 \times 2.82 \times 100}$$

$$= 0.906$$

$$\text{A.V} = \text{FFA} \times 2$$

$$= 0.906 \times 2$$

$$= 1.8$$

SAPONIFICATION VALUE

V_0 = Volume for blank test, 25.1 mL

V_1 = Volume for determination

Sample weight = 2 g

$$S.V = \frac{\text{Molecular weight for KOH} \times M \text{ of HCL used} \times (V_0 - V_1)}{\text{Sample weight}}$$

Casa 5

V_1 = 12.5 mL

$$S.V = \frac{56.1 \frac{g}{mol} \times 0.5 \frac{mol}{dm^3} \times (25.1 - 12.5) mL \times 1 \frac{dm^3}{1000 mL} \times 1000 \frac{mg}{g}}{2g}$$

$$= 176.72$$

Casa 101

V_1 = 12.6 mL

$$S.V = \frac{56.1 \frac{g}{mol} \times 0.5 \frac{mol}{dm^3} \times (25.1 - 12.6) mL \times 1 \frac{dm^3}{1000 mL} \times 1000 \frac{mg}{g}}{2g}$$

$$= 175.31$$

IODINE VALUE

V_0 = Volume for blank test, 47.1 mL

V_1 = Volume for determination

W = Sample weight, 0.4 g

C = Concentration of Sodium Thiosulphate used, 0.1M

$$I.V = \frac{126.9 \times C \times (V_0 - V_1)}{10 \times W}$$

Casa 5

$V_1 = 20.5$ mL

$$\begin{aligned} I.V &= \frac{126.9 \times 0.1 \times (47.1 - 20.5)}{10 \times 0.4} \\ &= 84.39 \end{aligned}$$

Casa 101

$V_1 = 21.2$ mL

$$\begin{aligned} I.V &= \frac{126.9 \times 0.1 \times (47.1 - 21.2)}{10 \times 0.4} \\ &= 82.17 \end{aligned}$$

SPECIFIC GRAVITY

W_0 = empty bottle weight, 9.5861 g

W_1 = Weight of bottle with oil

W_2 = Weight of bottle with water, 17.6563 g

$$\text{S.G} = \frac{(W_1 - W_0)}{(W_2 - W_0)}$$

Casa 5

W_1 = 17.3165 g

$$\begin{aligned} \text{S.G} &= \frac{(17.3165 - 9.5861)}{(17.6563 - 9.5861)} \\ &= 0.9578 \end{aligned}$$

Casa 101

W_1 = 17.3160 g

$$\begin{aligned} \text{S.G} &= \frac{(17.3160 - 9.5861)}{(17.6563 - 9.5861)} \\ &= 0.9578 \end{aligned}$$

VISCOSITY

Use spindle S03, at 10 rpm in the room temperature, 25°C

Casa 5

637.9cP = 6.379st

Casa 101

657.6cP = 6.576st

REFRACTIVE INDEX

Casa 5 = 1.4773

Casa 101 = 1.4784

pH VALUE

Casa 5 = 7.27

Casa 101 = 7.2

APPENDIX D**CALCULATION OF PERCENTAGE OF OIL EXTRACTED FROM SOXHLET
EXTRACTION AND SOLID-LIQUID EXTRACTION****CALCULATION****PERCENTAGE OF OIL EXTRACTED**

= Kernel weight (g) / Oil obtain (g) x 100

Soxhlet Extraction

Kernel weight = 36.2 g

Weight of extracted oil = 14.8 g

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{14.8}{36.2} \times 100 \\ &= 40.88\%\end{aligned}$$

Solid-Liquid Extraction

Kernel weight = 18.0 g

Weight of extracted oil = 7.8 g

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{7.8}{18.0} \times 100 \\ &= 43.3\%\end{aligned}$$

APPENDIX E**CALCULATION OF PERCENTAGE OF OIL EXTRACTED USING DEHULED
CASTOR SEED AND CRUSHED CASTOR SEED****CALCULATION****PERCENTAGE OF OIL EXTRACTED**

= Kernel weight (g) / Oil obtain (g) x 100

Dehuled Castor Seed

= Kernel weight (g) / Oil obtain (g) x 100

Kernel weight = 36.2 g

Weight of extracted oil = 14.8 g

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{14.8}{36.2} \times 100 \\ &= 40.88\%\end{aligned}$$

Crushed Castor Seed

= Seed weight (g) / Oil obtain (g) x 100

Seed weight weight = 50.0 g

Weight of extracted oil = 27.0 g

$$\begin{aligned}\% \text{ of oil extracted} &= \frac{27.0}{50.0} \times 100 \\ &= 54.0\%\end{aligned}$$