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

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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 kimia nya 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 diperoleh 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 neutralkan dan dilunturkan. Sifat minyak castor di bandingkan dengan ASTM kualiti. Kesimpulannya, minyak mentah dan minyak ditapis dan ditulenkan 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.

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, Japtropha 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 Pharmocoepia. 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 '000 t	1991 000 t	1992 '000 t	1993 '000 t	1994 '000 t	1995 '000 t	1996 '000 t	1997 '000 t	1998 '000 t	1999 '000 t	2000 '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 alkoxylated 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)

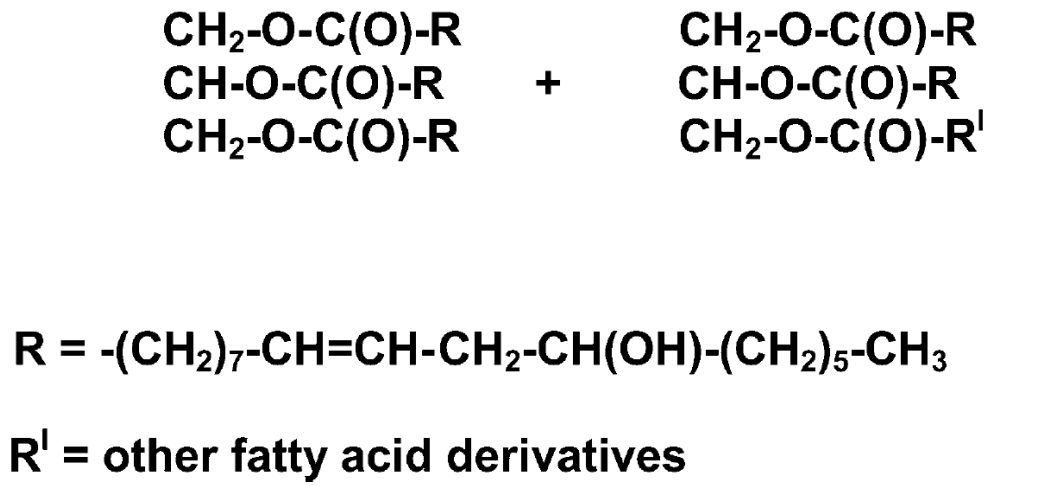

Figure 2.1: Equation showing the constitution of castor oil (Ogunniyi, 2006)

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**)**.

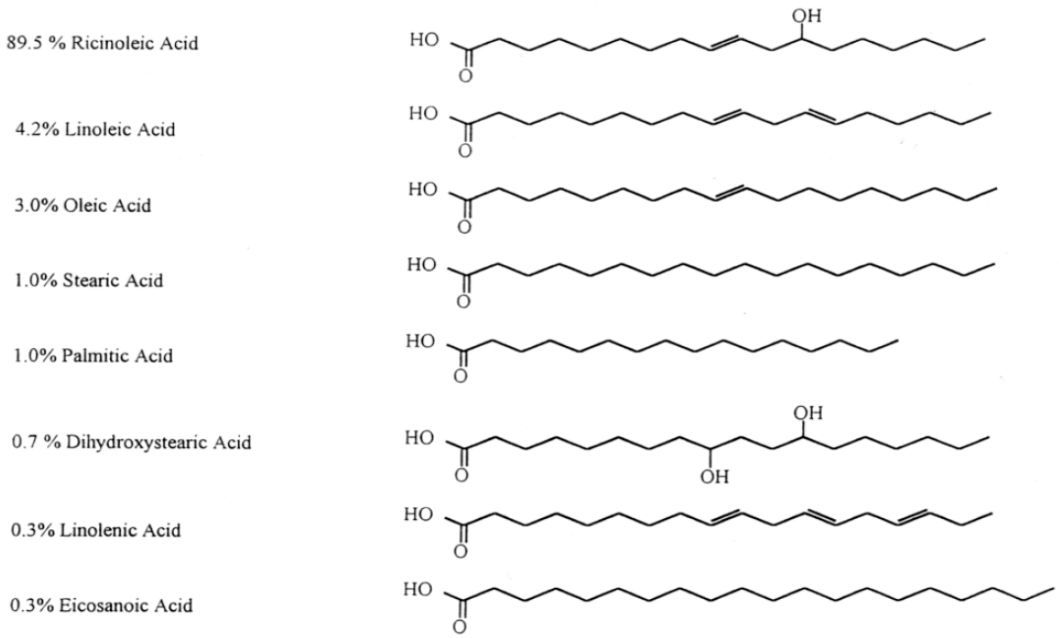

Figure 2.2: Composition of castor oil fatty acids

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_2SO_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_5, $POCl_3$	Halogenated castor oils
	Alkoxylation	Ethylene and/or propylene oxide	Alkoxylated castor oils
	Esterification	Acetic-, phosphoric-, maleic-, phthalic anhydrides	Alkyl and alkylaryl esters, phosphate esters
	Sulfation	H_2SO_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 save 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 of 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.

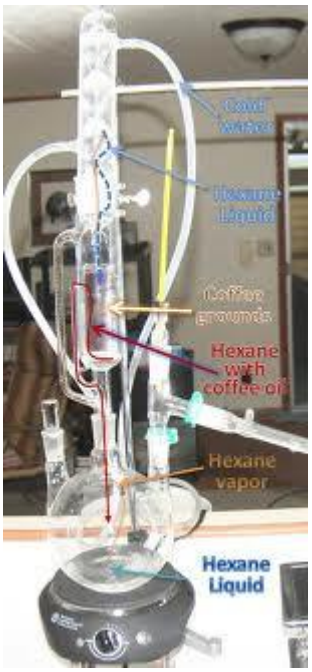

Figure 3.2: Laboratory soxhlet extractor

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.

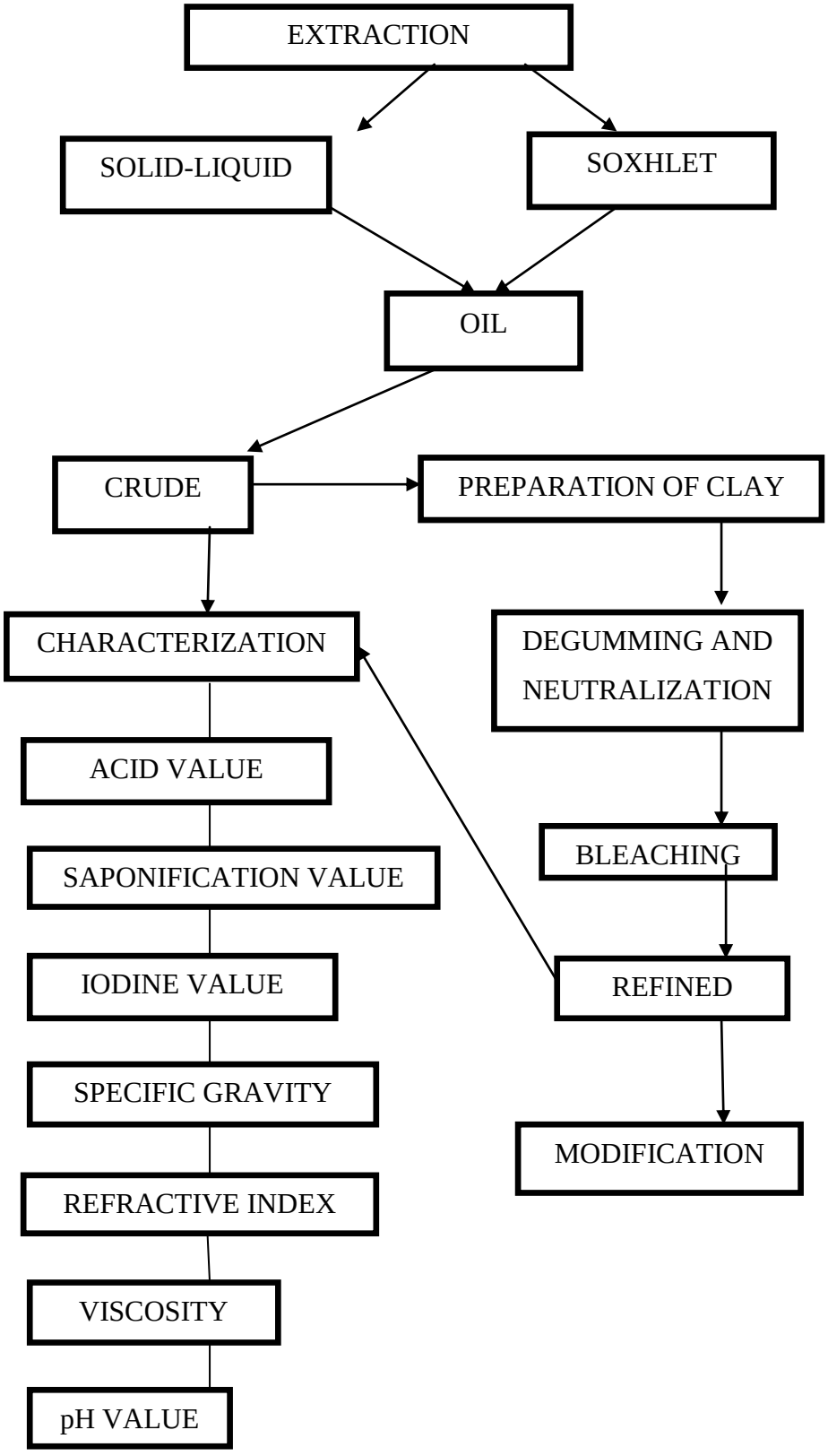

Figure 3.4: Flow of the experimental work

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.

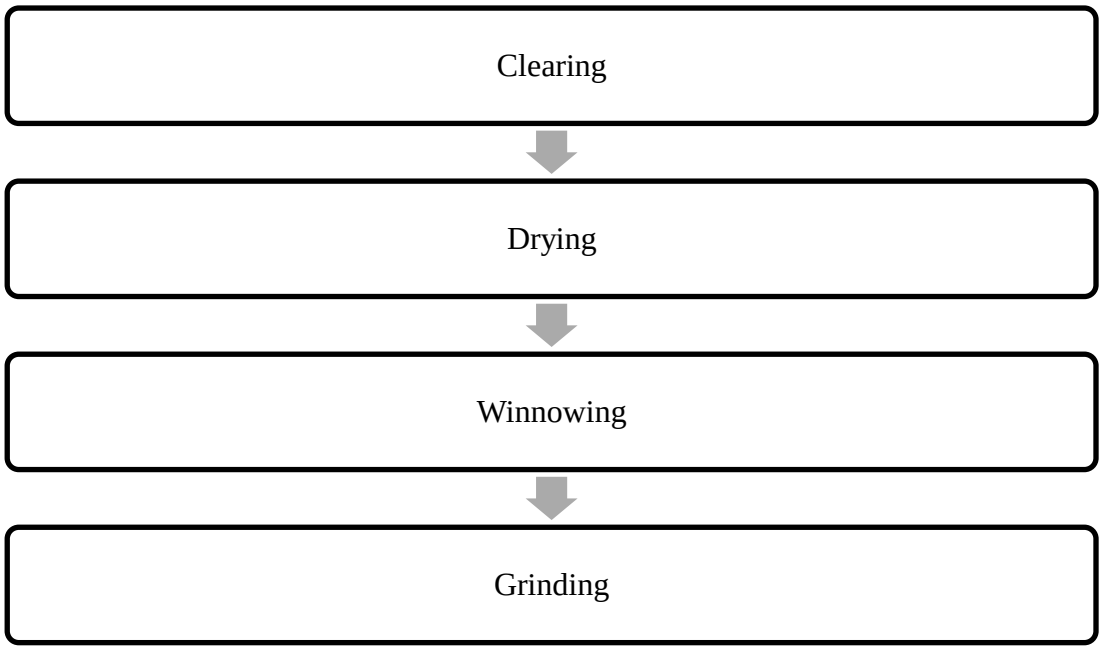

Figure 3.5: Flow chart of castor bean processing

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.

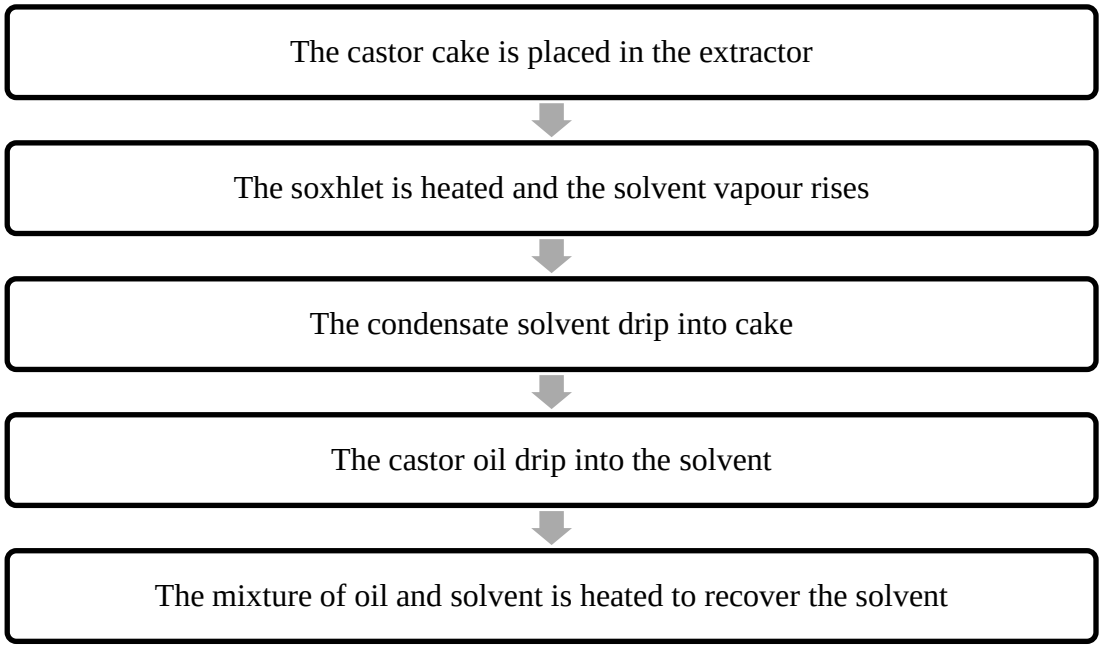

Figure 3.6: Flow chart of operation of soxhlet extractor

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.

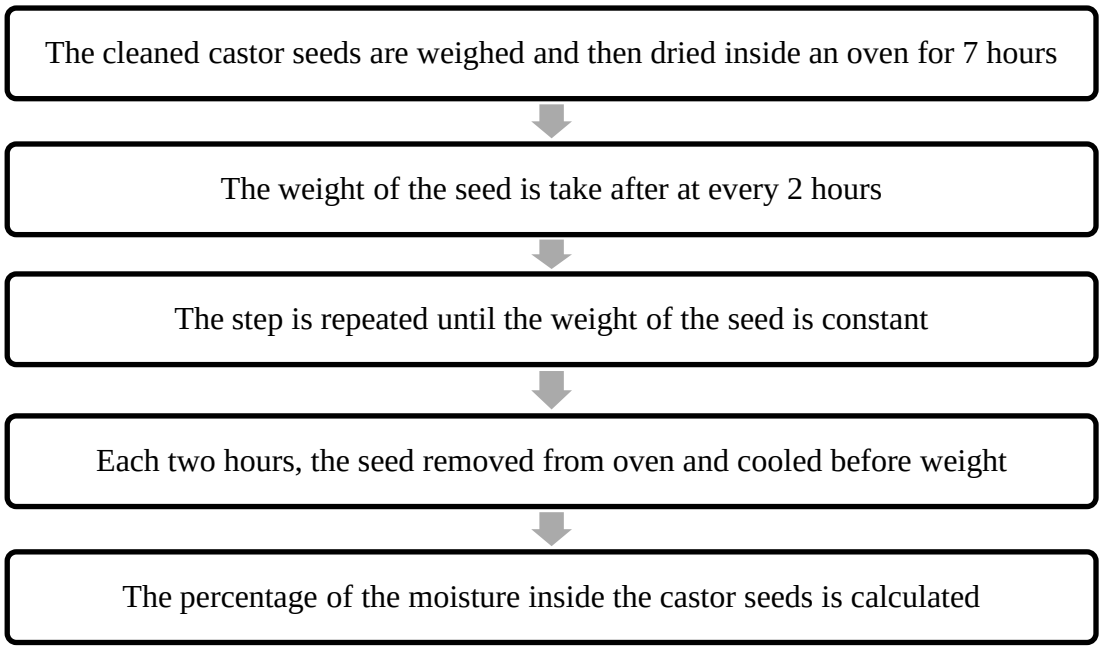

Figure 3.7: Flow chart of determining the moisture content

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.