PRODUCTIVITY AND EFFICIENCY OF BIODIESEL FROM ROSELLE SEEDS FOR RENEWABLE FUEL

KITTAPAS HOMRARUEN

A DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF DOCTOR OF ENGINEERING

PROGRAM IN ENERGY AND MATERIALS ENGINEERING

(INTERNATIONAL PROGRAM)

FACULTY OF ENGINEERING

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Name-Surname	Mr.Kittapas Homraruen
Program	Energy and Materials Engineering
Dissertation Advisor	Mr. Winai Chanpeng, Ph.D.
Academic Year	2021

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June 12, 2020

ชื่อวิทยานิพนธ์	ผลผลิตและประสิทธิภาพของไบโอดีเซลจากเมล็ดกระเจี๊ยบแดงสำหรับเชื้อเพลิงทดแทน
ชื่อ-นามสกุล	นายกฤตภาส หอมระรื่น
สาขาวิชา	วิศวกรรมพลังงานและวัสดุ
อาจารย์ปรึกษา	อาจารย์วินัย จันทร์เพ็ง, วศ.ด.
ปีการศึกษา	2564

บทคัดย่อ

กระเจี๊ยบแดง *(Hibiscus sabdariffa L.)* เป็นพืชผลที่สำคัญในประเทศเขตร้อนและกึ่งเขตร้อน ปกติเกษตรกรจะเก็บเกี่ยวเฉพาะกลีบดอกเพื่อไปจำหน่าย แต่เมล็ดของกระเจี๊ยบแดง เกษตรกรจะเก็บไว้บางส่วนใช้เพื่อขยายพันธ์ของรุ่นต่อไป เนื่องจากมีเมล็ดกระเจี๊ยบแดงเหลือทิ้งเป็นจำนวนมาก และภายในเมล็ดกระเจี๊ยบแดงมีปริมาณน้ำมันสูง ซึ่งสามารถมาสกัดเป็นน้ำมัน และนำไปผ่านกระบวนการทางเคมีจึงได้น้ำมันไบโอดีเซล การวิจัยครั้งนี้จึงมีวัตถุประสงค์ ก) เพื่อศึกษาคุณสมบัติสารระเหย คาร์บอนคงที่ และปริมาณเถ้าของเมล็ดกระเจี๊ยบแดงในระดับความชื้นที่ต่างกัน ข) เพื่อวิเคราะห์หากรดไขมันของเมล็ดกระเจี๊ยบแดงในระดับความชื้นที่ต่างกัน ค) เพื่อตรวจสอบน้ำมันไบโอดีเซลที่ได้จากเมล็ดกระเจี๊ยบแดงว่าตรงตามมาตรฐานสากล

เริ่มจากนำเมล็ดกระเจี๊ยบแดงที่มีระดับความชื้นต่างกัน 3 ระดับ คือ 8% 10% และ 12% ไปทดสอบในห้องทดลองโดยใช้เครื่องมือเพื่อศึกษาหาสารระเหย คาร์บอนคงที่ และปริมาณเถ้า ในการหากรดไขมันของเมล็ดกระเจี๊ยบแดง นำน้ำมันที่สกัดจากเมล็ดกระเจี๊ยบแดงที่ระดับความชื้น 3 ระดับมาผ่านกระบวนการโดยใช้วิธีเทคนิคแก๊สโครมาโทกราฟี (GC-FID) ในการตรวจสอบคุณสมบัติไบโอดีเซล น้ำมันที่สกัดจากเมล็ดกระเจี๊ยบแดงจะถูกนำไปตรวจสอบจุดไหลเท ความเป็นกรด ความหนืด จุดวาบไฟ และ ความหนาแน่นเพื่อนำไปเปรียบเทียบมาตราฐานสากล

ผลการวิจัยพบว่าเมล็ดกระเจี๊ยบมีความชื้น 8 ถึง 12 เปอร์เซ็นต์ ที่ปริมาณ 1274-1323 มล. ต่อ 0.0468 กรัม ในน้ำมัน สารระเหย 69.83-73.01% คาร์บอนคงที่ 16.07-17.82% เถ้า 6.92-8.47% ความชื้นของเมล็ดกระเจี๊ยบแดง 8-12% ปาล์มเมท 20.81-22.46% กรดสเตียริก 4.83-5.04% กรดโอเลอิก 38.80-47.73% กรดลิโนเลอิก 21.69-33.33% และกรดจีลิโนเลอิก 2.16-2.71% ต่อจากนั้นผลการวิเคราะห์คุณสมบัติไบโอดีเซล ในแง่ของจุดไหลเท ความเป็นกรด ความหนืด จุดวาบไฟ และความหนาแน่นเป็นไปตามมาตรฐานไบโอดีเซล ดั้งนั้นน้ำมันเมล็ดกระเจี๊ยบสามารถนำมาใช้เป็นเชื้อเพลิงทดแทนได้

คำสำคัญ: เมล็ดกระเจี๊ยบแดง ความชื้น คุณสมบัติของเชื้อเพลิง ไบโอดีเซล

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ABSTRACT

Roselle *(Hibiscus sabdariffa L.)* is an important crop in tropical and subtropical countries. Normally, farmers harvest only its sepals or petals for selling purposes. They keep some seeds for propagating the next crop, so there are a lot of seeds thrown away. As Roselle seeds are high in oil content, the researcher is interested in making use of Roselle seeds and exploiting this quality. The objectives of this research were: a) to examine the properties of volatile matter, fixed carbon and ash content in different levels of moisture in Roselle seeds; b) to investigate fatty acids in different levels of moisture in Roselle seeds; c) to verify the properties of biodiesel produced from Roselle seeds in accordance with international standards.

Roselle seeds with three levels of moisture, namely 8%, 10%, 12% were tested by particular laboratory instruments to examine the properties of volatile matter, fixed carbon and ash content. To investigate fatty acids, oil extracted from Roselle seeds at these three moisture levels was tested by gas chromatography (GC-FID) technique. To verify the properties of biodiesel oil from Roselle seeds in accordance with international standards, oil extracted from Roselle seeds was tested to find the properties of pour point, acid value, viscosity, flash point, and density.

The research results showed that Roselle seeds with a moisture of 8 to 12 percent at the volume of 1274-1323 ml. per 0.0468 g in oil had volatile matter of 69.83-73.01%, fixed carbon of 16.07-17.82%, ash of 6.92-8.47%. Moreover, they had palmitate 20.81-22.46%, stearic acid 4.83-5.04%, oleic acid 38.80-47.73%, linoleic acid 21.69-33.33%, and 2.16-2.71% g-linoleic acid. In terms of the properties of pour point, acidity, viscosity, flash point, and density, it was found that they met the biodiesel standards. These findings confirm that Roselle seed oil can be used to produce an alternative fuel.

Keywords: roselle seeds, moisture, fuel properties, biodiesel

Acknowledgments

I would like to express my sincere gratitude to my thesis advisor Dr.Winai Chanpeng for the valuable guidance and encouragement which helped me in all times of my research.

I would like to thank the thesis committees, Assistant Professor Dr. Phairoach Chunkaew, Assistant Professor Dr. Phanu Prathumnoppharat, Assistant Professor Dr. Kiattisak Saengpradit, and Assistant Professor Dr. Boonrit Prasartkaew for their valuable comments and helpful suggestions, have provided me tremendous suggestions and motivation to think carefully on the research ideas and methodology throughout the dissertation duration process. Furthermore, I thank the Rajamangala University of Technology Tanyaburi for the tremendous financial supports and flexibility during the several previous years of my educations.

I am most thankful for my family who always supports and encourages me in every endeavor in my life. Without their love patience and encouragement, I will not be who I am today.

Kittapas Homraruen

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CHAPTER 1

INTRODUCTION

1.1 Background and Importance

Although petrochemicals, coal, hydropower and nuclear power are the main energy sources around the world, [1] the petroleum reserves are limited. The demand for fossil fuels around the world continues to increase. The loss of fossil fuels affects the environment, hence a springboard for researchers to find alternative energy [2]. Reducing the overall volume of these reserves has encouraged researchers to develop alternative energy sources [3]. The production of biomass gas condenses the formation of aerosols during the gasification process. Bitumen, an important contaminant in gas production, causes problems [4].

According to the above information, it is better to use bio-oil to produce alternative fuels. Fuels produced from biomass could be a replacement of fossil fuels [5] and biomass fuels are perceived as environmentally friendly when compared to conventional diesel because biofuels have significantly lower carbon monoxide and hydrocarbon emission values [6]. Bio-oil can be obtained from palm oil and tung oil industry. The production process in Thailand causes a lot of waste pollution [7]. Due to economic and environmental concerns, the demand for a substitute fuel supply has increased. Biodiesel is one of such supply and its production has now become a worldwide industry. And till date, Thailand still imports oil for industrial and population development, both the public and private sectors [8].

The scientific name of Roselle is Hibiscus sabdariffa L. The common name is Jamaican Sorrel, it belongs to the family of Malvaceae, the different name; Keng Kheng Orange, Sour roselle, Roselle, Keng Kheng vegetables, Keng Keng. Its botanical characteristics are as thus: it is a shrub of 50-180 cm with a variety of magenta leaves and a palm trunk, three or five lobes of similar length and width 8-15 cm. The flowers have axillary pink petals and are yellow at the center. There are magenta pollen tubes connected to a dry fruit covered with a juicy red sepal [9]. After harvest, farmers will

keep a small number of seeds for planting in the future. When compared with others, they are remaining agricultural seeds. Finding the most appropriate drying method to obtain the optimum moisture content of the plants depends on the differences in the shape of the seed, season, and geographical location [10].

So, of particular interest for this experimental research is to study Roselle seeds from Amphoe Doembang Nangbuat, Changwat Suphanburi in the central region of Thailand, through the method of drying, in order to find out the different moisture values to compare the volume of the extracted oil. The next is, an investigation of the relationship of the moisture using thermal analysis will be undertaken to identify the organic compounds as well as the carbon, and ash contained in the Roselle seeds. To analyze the basic properties of oil quality characteristics; the ASTM and DIN EN standards are used, such as the Iodine value, specific gravity, thermal value, flash point, and viscosity. The chemical composition of bio-oil produced from Roselle seeds was qualitatively analyzed using the Gas Chromatography - Flame Ionization Detector (GC-FID) and analyzed for the amount of heat from combustion reactions to understand the energy obtained from the bio-oil of Roselle seeds.

1.2 Objectives of the Study

1.2.1 To examine the properties of volatile matter, fixed carbon and ash content in different levels of moisture in Roselle seeds.

1.2.2 To investigate fatty acids in different levels of moisture in Roselle seeds.

1.2.3 To verify the properties of biodiesel produced from Roselle seeds in accordance with international standards.

1.3 Scope and Limitation of the Study

1.3.1 The analysis of the effects of moisture on Roselle seeds.

1.3.2 The analysis of the properties of oil content, volatile matter, fixed carbon, ash content, and fatty acids.

1.3.3 The examination of the properties of biodiesel produced from Roselle seeds in the form of Transesterification.

1.4 The expected benefits to be derived

1.4.1 The knowledge of the effect of moisture on Roselle seeds.

1.4.2 The knowledge of the properties of oil content, volatile matter, fixed carbon, ash content, and fatty acid content.

1.4.3 The knowledge of the properties of biodiesel produced from Roselle seeds in a transesterification form.

CHAPTER 2

RELATED THEORY AND LITERATURE REVIEW

2.1 Bright future for truly sustainable biofuels

To respond to concerns about the long- term maintainable production of large-scale biofuel, the EU recommended a mandatory sustainability criterion for biofuels in 2009. With this criterion, the EU has set a major example worldwide, a policy for a more reliable use of biofuel with little or no harm to the environment, which not only impacts the biofuel sector, but other sectors as well.

This long-term maintainable criterion helps to solve the problems of direct risks from biofuels, leading to a minimum GHG saving compared to fossil fuels, and protecting ecological diversity and highly carbonated stock lands, like wetlands and peatlands. However, the criteria stipulated by the EU have shortcomings. They focus more especially on the direct but not the indirect impacts of biofuel production and sustainability.

In the year 2012, the European Commission published a proposal with objectives to address ILUC to cut-down on conventional biofuels at 5% of road transport fuel and encouraging biofuels from alternate sources like wastes, residues and cellulosic material [11].

Figure 2.1 Residue materials like roselle seeds and nutshells could serve as feedstock.

SOURCE: www.saraphishop.blogspot.com and www.nanagarden.comproduct 250236

2.2 Roselle

The scientific name of Roselle is Hibiscus sabdariffa L. The common name is Jamaican Sorel, it belongs to the family of Malvaceae, different name: Roselle, Sour roselle, Keng Kheng vegetables, Keng Kheng Orange, Taelg devout Orange. Its botanical characteristics are as thus: it is a shrub of 50-180 cm with a variety of magenta leaves and a palm trunk, three or five similar lobes of length and width 8-15 cm. The flowers have axillary pink petals and yellow at the center. There are magenta pollen tubes connected to a dry fruit covered with juicy red sepals.

Figure 2.2 Roselle sample

2.3 Biodiesel

Biodiesel is defined technically as “the mono alkyl esters of long fatty acids obtained from renewable lipids of feedstock such as vegetable oils or animal fats, used for compression and ignition in diesel engines” [12]. In its simplest form, biodiesel is a renewable fuel which is manufactured from methanol and vegetable oil, animal fats, and recycled cooking fats [13].

The term “biodiesel” itself is often misunderstood, hence misused as well. In clearer terms, it actually refers to 100% pure fuel (B100) that meets the above definition

and specific standards as given by the American Society of Testing and Materials (ASTM) International (D6751) [14].

It is equally also often used to describe the combination of biodiesel with petroleum diesel. These combinations are usually referred to as; “B2,” “B5,” “B20,” etc. In this case, the number represents the percent of biodiesel used. Commonly, biodiesel is produced through a process called “transesterification,” which involves changing the chemical properties of the oil by using methanol [15].

Biodiesel is a form of vegetable oil - or animal fat-based oil consisting of long-chain alkyl (methyl, ethyl, or propyl) esters. Biodiesel is obtained typically by chemically reacting lipids (e.g. vegetable oil, soybean oil [16] and animal fat (tallow [17] [18])) with an alcohol producing fatty acid esters. Biodiesel blends can also be used as heating oil.

The United States of America National Biodiesel Board also has a technical definition of "biodiesel" as a mono-alkyl ester [19].

A combination of biodiesel and conventional hydrocarbon-based diesel products are commonly distributed for sale in the retail diesel fuel market. Majority of the world uses a system called the "B" factor to ascertain the amount of biodiesel in any fuel mix [20].

100% biodiesel is marked as B100

20% biodiesel, 80% Petro diesel is branded as B20

5% biodiesel, 95% Petro diesel is marked as B5

2% biodiesel, 98% Petro diesel is branded as B2

A combination of biodiesel lower than 20% can be used in diesel equipment with little or minor adjustments [21], although some producers do not extend the warranty coverage if the equipment is damaged by these blends. The B6 to B20 blends are covered by the ASTM D7467 specification [22]. Biodiesel can also be used in its pure form (B100), but may require the adjustments of certain engines to avoid

maintenance and performance problems [23]. Blending B100 with petroleum diesel may be accomplished by:

Mixing the content in tanks at manufacturing point prior to delivery to tanker truck.

Splash mixing in the tanker truck by adding specific percentages of biodiesel and petroleum diesel.

The simultaneous mixing of two components arriving at tanker truck at the same time.

The mixing of petroleum diesel and biodiesel pump meters set to X total volume and transfer pump pulls from two points and the mixing is complete on leaving the pump.

Figure 2.3 Sample of biodiesel

2.4 The Transesterification process

This is a process of chemical reaction utilized for the conversion of vegetable oil to biodiesel. During this process, vegetable oil is chemically reacted with an alcohol - like methanol or ethanol in the presence of a catalyst like lye. The result of the

chemical reaction will be the break down of various components of the vegetable oil to form new compounds.

The triglycerides are changed into alkyl esters, which is the chemical name of biodiesel. The change depends on which chemical is used in the reaction. If methanol is used, methyl esters are formed, but if ethanol is used, then there is the formation of ethyl esters. Both of these compounds have different chemical combinations. In the chemical reaction alcohol takes the place of glycerin.

The glycerin derived during the transesterification process is released as a byproduct of the chemical reaction. Glycerin will either settle at the bottom or at the surface of the vessel depending on its phase, and then it can easily be separated by centrifuges in a process called transesterification [24]. However, the production of biodiesel from other sources or by other methods may require a much slower acid catalyst [25], because it is the only predominant method for production for commercial purposes. The catalyzed-base transesterification process is described below. Triglycerides (1) reacts with an alcohol such as ethanol (2) to give ethyl esters of fatty acids (3) and glycerol (4).

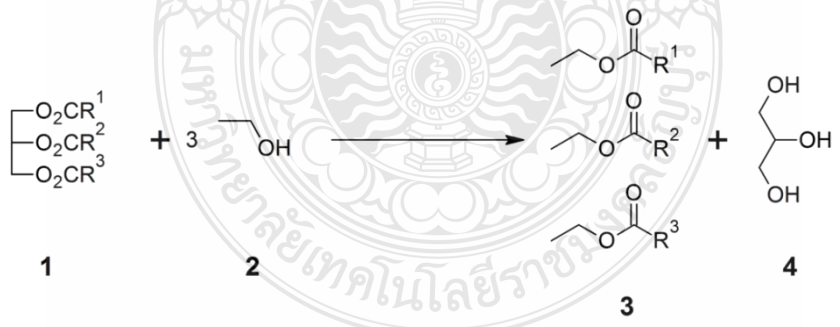


Figure 2.4 Chemical reaction process of transesterification.

2.5 Potassium Hydroxide (KOH)

Potassium hydroxide (KOH), solid and colorless, odorless, non-flammable and non-volatile, with a molecular weight of 56.10 and melting point of 360°C, with

specific gravity of 2.04, can react with carbon dioxide in the air and produce potassium carbonate and potassium hydroxide.

Potassium hydroxide (KOH) of electricity through brine potash and potassium chloride is similar to a method of producing sodium hydroxide which is a material used for the production of mild soap and liquid soap, paper and synthetic fibers. Batteries are also used for the production of alkaline cells (Alkaline cell) as swell as potash and potassium carbonate purity in the glass industry. Potassium hydroxide (KOH) is strongly alkaline and corrosive to tissues, but less than the effect of sodium hydroxide [26].

Figure 2.5 Potassium Hydroxide sample

SOURCE: www.worldchemical.co.th

2.6 Sodium Hydroxide (NaOH)

Sodium hydroxide (NaOH) has a solid white crystalline appearance. It is odorless, non-flammable and non-volatile with a molecular weight of 40.01and a melting point of 318°C, pH, specific gravity of 2.13 as well as soluble in water, heat and smoke or aerosol. It can bleed easily when it reacts with moisture, acid or a salt of the substance, and react with the fatty acid soap. It also reacts with carbon dioxide in the air which can easily cause sodium.

This material is obtained by passing electric current in the water and in sodium chloride. The sodium ions change to positive electrode (Cathode) with a sheet of

asbestos, hence prevent chlorine, then it reacts with water to give out hydrogen gas and sodium hydroxide. Sodium hydroxide (NaOH) is a corrosive tissue and violently reacts with proteins and fats, causing the soft, gelatinous or gelatin and soap. The tissue is destroyed or torn down, then its destruction may continue for several days. It is hard to rinse out [27].

Figure 2.6 Sodium Hydroxide sample

SOURCE: www.siamchemi.com

2.7 Alcohol

Alcohol is an organic compound that is a derivative of hydrocarbons where H is replaced by -OH functional groups with the general formula R-OH being a functional hydroxyl group (hydroxyl group; -OH).

Table 2.1 The Alcohol Structure

Formula structure	Common name
CH3OH	methyl alcohol
CH3CH2OH	ethyl alcohol
CH3CH2CH2OH	propyl alcohol
CH3CH2 CH2CH2OH	butyl alcohol
CH3CH2 CH2CH2CH2OH	penticton alcohol

The boiling point of alcohol is increased by increasing the number of carbon atoms. When the number of carbon atoms is augmented, the molecular weight is increased. The bond between the molecules becomes higher because alcohol is a polar molecule. The London force has both the strength and attraction between the poles. The bond between molecules and among -OH is hydrogen bond as well as the bond between the molecules of alcohol.

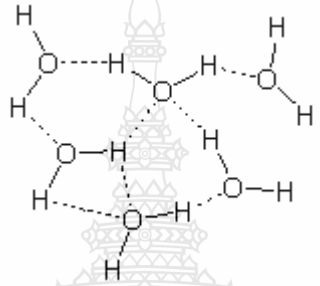


Figure 2.7 The hydrogen bond between water molecules

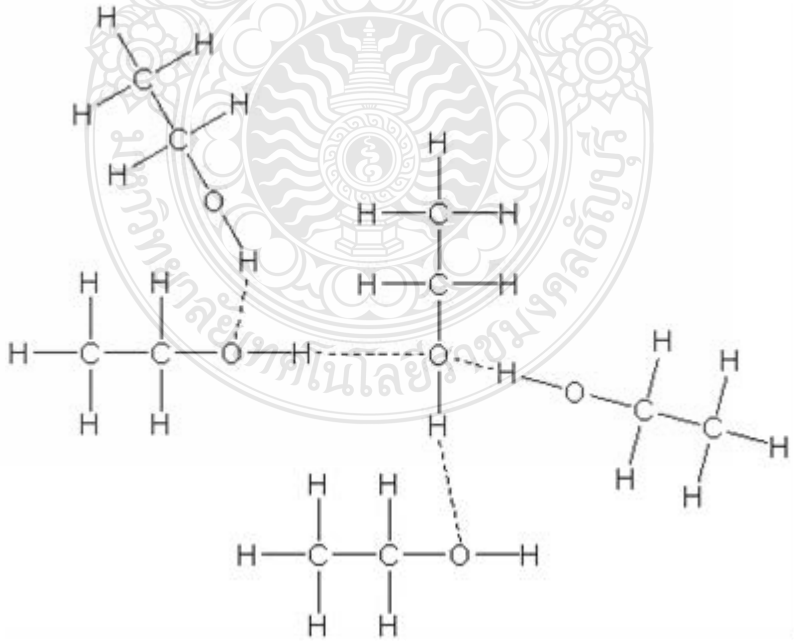


Figure 2.8 The hydrogen bond between the molecules of alcohol

The boiling point of alcohol is higher than that of alkane with the same number of atoms of carbon. The alkane molecules are polar and the only force is London.

2.8 Methanol

This alcohol has a minimal number of carbon atoms prepared by burning wood at high temperatures in air-free conditions. In industries, it is prepared from a reaction between carbon monoxide and hydrogen under high pressure and temperature with metal oxides, such as Fe2O3, ZnO / Cr2O3 catalyst. The equation is

$$CO + H2 \xrightarrow{\text{----------}} CH3OH$$

Methanol

2.9 Ethanol

This alcohol is obtained from the fermentation of sugars derived from fruits or starch from cereals in the absence of oxygen. The enzyme from yeast or bacteria accelerates the reaction, as seen in equation [28].

$$C6H12O6 \xrightarrow{\text{-----}} 2CH3CH2OH + 2CO2$$

Ethanol

2.10 Titration

2.9.1 Chemicals used in titration

2.9.1.1 Alkaline potassium hydroxide or sodium hydroxide

2.9.1.2 Roselle seeds oil.

2.9.1.3 Isopropyl alcohol or ethanol propanol

2.9.1.4 Phenol naphtha lean

2.9.2 The process of titration

2.9.2.1 Soluble alkali 50 g in 500 ml of distilled water, so it has an alkaline solution at a concentration of 10%.

2.9.2.2 Dissolved alkaline solution with volume of 5 ml of distilled water, 500 ml, so it has an alkaline solution at a concentration of 0.1%, which is the desired concentration.

2.9.2.3 Soluble ingredients in water after boiling. Add repellant and mix with 1 ml of isobutane propellants Expo 10 ml alcohol.

2.9.2.4 Then drop five drops of phenol naphthalene in a glass jar and stir to combine with visit Phu heater in order to help melting into each other well.

2.9.2.5 In the solution, drops two drops using a dropper and count the number of drops of the oil until it changes color into purple and pink. Each drop should have a time interval of about one minute. Shake thoroughly for the drops to be so reactive.

2.9.2.6 Once the water turns purple and pink, stop dropping and count 30 seconds. The constant color is purple and pink. To stop the drops, count to calculate the amount of alkaline reaction to the recipe below. If the color has come back clear, drop further drops and then watch for changes; 2-5 drops of color will turn purple, pink constant.

2.9.2.7 The calculation of the amount of water used in the reaction is 1 drop of 0.05 ml dropper or 20 drops equal 1 ml [29].

Sodium hydroxide (NaOH)

Formula

$$(\text{Number of drops} \times 0.05) + 3.50 \qquad (2.1)$$

For example, if the solution to 50 drops

50 x 0.05 + 3.50 = 6.00 ml, Or 6.00 g.

So, the amount of sodium hydroxide needed per one liter oil = 6.00 g.

Potassium hydroxide (KOH)

Formula: (Number of drops x 0.05) + 5.00 (2.2)

For example, if the solution to 50 drops

50 x 0.05 + 5.00 = 7.50 ml, Or 7.50 g.

So, amount of Potassium hydroxide needed per one liter oil = 7.50 g.

2.11 Literature review

Hussain Al-Wandaw et al. [30] in their study of the Chemical analysis of adult Roselle seeds (HibiscussabdariffaL.) found that the seeds contain protein (25.20%) and fat (21.10%). Detects and determines the amount of eight amino acids. First, a minimal number of amino acids were found, which are; triplofen, valine, isozyme, and threonine with a chemical score equal to 45.33,52.54,55.34 and 58.80 respectively, while the highest found that the essential amino acids include leucine, lysine, and phenylalanine. For most oleic acid the dominant fatty acids are followed by Palmitic and stearic acid. The analysis of the components revealed that K, Na, Mg, and Ca are the main components. Gossypol is a mere trace.

Table 2.2 Chemical Composition of Roselle Seeds [a]

component	%[b] (dry wt basis)
protein ($N \times 6.25$)	25.20
lipids	21.10
crude fiber	16.30
starch[c]	2.25
ash	5.19
total carbohydrate[d]	26.64
moisture	5.57
gossypol: free	trace
bound	trace

[a] Mature whole sun-dried seed. [b] The average of triplicate analysis.

[c] Percentage of starch in defatted and sugar-freed seed flour. [d] Total carbohydrate determined as 100 - (moisture + crude fiber + protein + lipids + ash).

Table 2.3 The composition of amino- acid [a] of mature whole Roselle seed

amino acid	g/16 g of nitrogen
Lys	5.56
His	1.87
Arg	10.75
Trp	0.68
Asp	10.16
Thr	2.94
Ser	4.37
Glu	23.45
Pro	3.29
Gly	5.08
Ala	4.09
Cys	2.50
Val	3.85
Met	1.35
Ile	3.21
Leu	6.31
Tyr	3.45
Phe	5.20

The data represent the average of duplicate analyses.

Table 2.4 The comparison of the Chemical Score Values of Essential Amino Acids of Roselle Seed with that of Okra Seeds

essential amino acids (EAA)[a]	roselle, Iraqi cultivar	okra	
		Emerald[b]	Ibtaira[b]
leucine	70.90	75.06	79.10
isoleucine	55.34	54.31	57.41
cysteine and methionine	67.54	71.93	76.34
valine	52.54	54.05	67.03
tryptophan	45.33	64.00	56.67
phenylalanine	92.86	76.43	70.36
lysine	83.00	117.91	133.13
histidine	89.05	84.76	87.62
threonine	58.80	60.00	70.00
tyrosine			

[a] EAA data for reference protein (whole egg) and the method used for its calculation in roselle and okra seeds were reported by Oshorne and Voogt (1978).
[b] AI-Wandawi (1983)

Table 2.5 Fatty Acid Composition [a, b] of Roselle (H,sobdariffa L.) Seed Oil

fatty acid	procedures[c]				mean	SD[d]
	A	B	C	D		
lauric	0.01	trace	0.02	0.01	0.01	0.005
myristic	0.22	0.40	0.36	0.30	0.32	0.08
palmitic	17.85	20.03	22.47	28.46	22.20	4.68
stearic	3.46	4.93	2.27	4.43	3.77	1.17
oleic	77.16	73.95	75.04	66.41	73.14	4.68
linoleic	0.05	0.23	0.64	0.06	0.25	0.28
arachidic	0.25	0.46	0.21	0.24	0.29	0.12

[a] The data above is for the average of duplicate analyses. [b]The values are expressed as a percent of total recovered fatty acids. [c] See the text for details of transesterification methods. [d] SD = standard deviation.

Table 2.6 The mineral compositions of H. sabdariffa Seed [a]

element	mg/100 g (dry wt basis)[b]
calcium	300
copper	<1
chromium	<1
iron	8.2
magnesium	580
manganese	3.5
nickel	1.0
potassium	1600
rubidium	<1
sodium	740
strontium	1.6
zinc	6.8

[a] Data are the average of three determinations on separate samples. [b] The flour of the seeds was defatted and dried for 2 hours at 105ºC before the analysis.

Juana Sanchez-Mendoza et al. [31] carried out a study of moisture content as was determined by the American Society of Agricultural Engineers (ASAE). The standard of this method ranges from ca. 13% to 25% w.b. He evaluated the condition of the modified seed to find the dimension of the following: harmony, density, true density, porosity, seed mass and static friction coefficient on various material surfaces. During the analysis of the physical properties of okra seeds, as related to the moisture content by the quadratic equation, the width of Mexican, Chinese and Sudan species increased by 10.9%, 7.8%, and 8.1% respectively; the length increased by 13.1%, 9.8% and 7.2% and its thickness also increased in 8.3%, 4.0%, and 13.7%. The seeds from Mexico are the smallest. The change of the sphere by increasing the amount of moisture, each specie has a single behavior. So, the Sudanese specie come close to the sphere, as opposed to the Mexican or Chinese species. The total density and true density for different humidity levels will decrease as the humidity increases. It was found that the porosity increased with increasing moisture content. The pores start at 52.9%, 53.3%, and 54.7% and increase to 57.7%, 55.1%, and 56.9% for varieties of Mexico,

China, and Sudan respectively. The mass of the seed increased by increasing the moisture content of the seeds from 21.85 to 81.00 g, 35.22 to 40.62 g, and 36.37 to 41.78 g for all three species mentioned above respectively. The evaluated species show differential behavior with regards to the relationship as well as the coefficient of static friction. This variable increases with the amount of moisture on the four surfaces, namely, concrete (0.32–0.34) galvanized steel (0.27–0.30), glass (0.25–0.30) and wood (0.23–0.30)

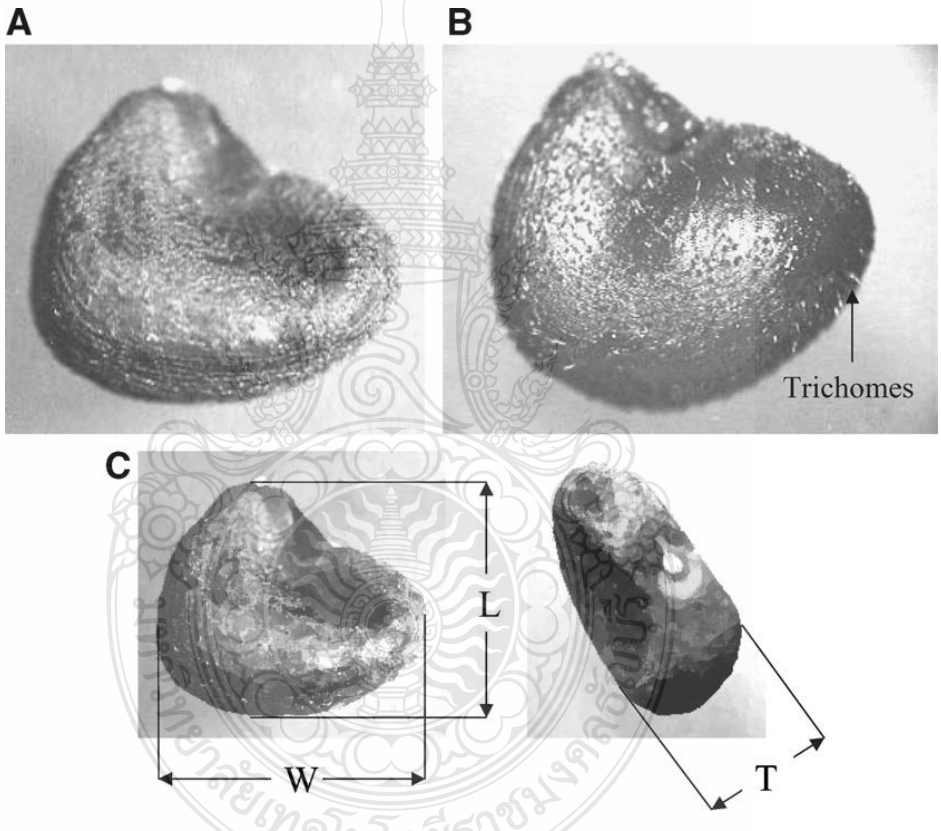


Figure 2.9 Roselle seed image at 7.5% (w.b.) moisture content (A) and at moisture saturation (B); (C) principal measured dimensions: L, length; W, width, and T, thickness.

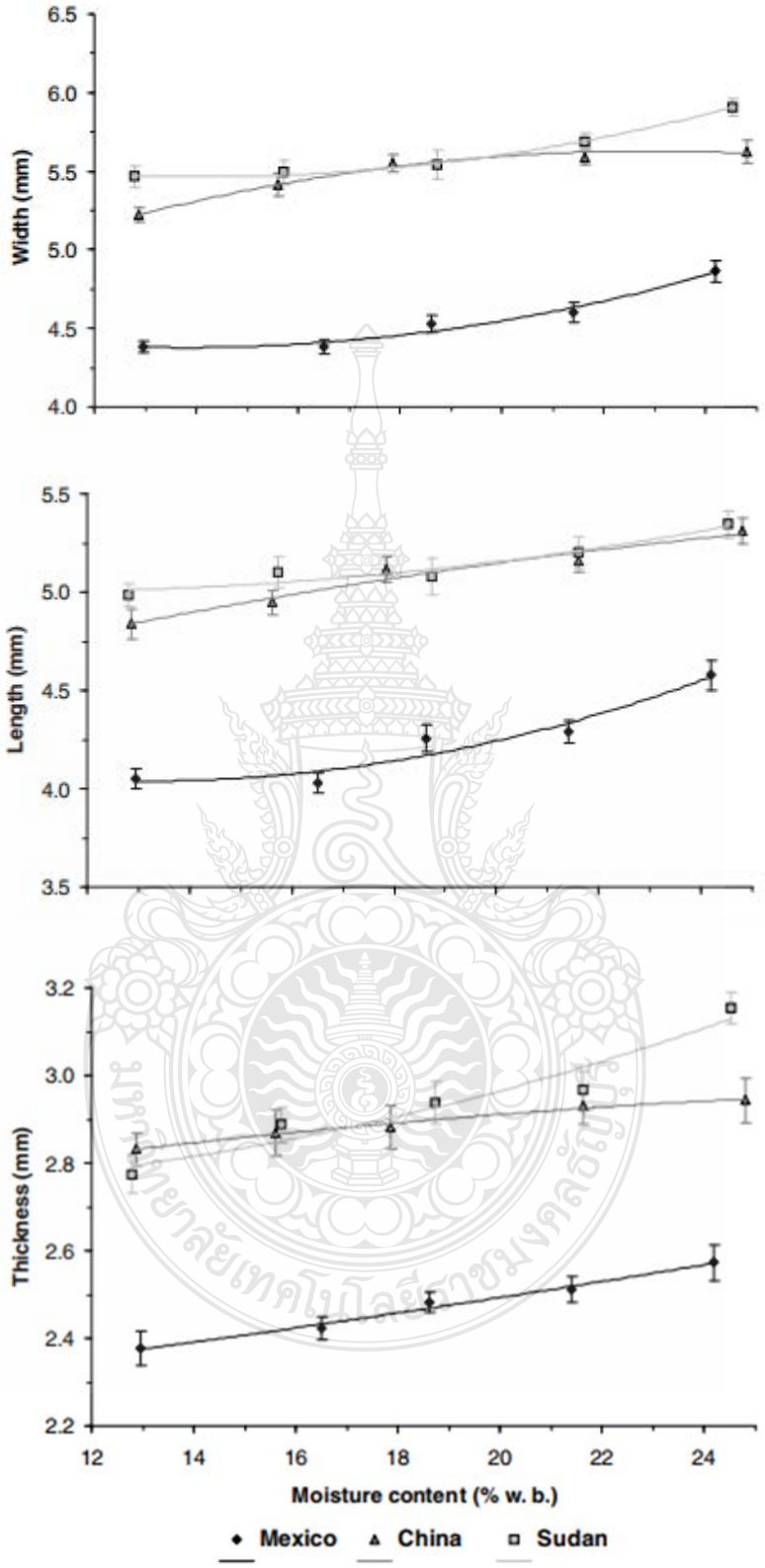


Figure 2.10 The effect of moisture content on the dimension Roselle seed.

Table 2.7 Coefficients β of the quadratic model and coefficient of determination R^2 for the dimensions of Roselle seed.

Cultivar	Parameter	Width (mm)	Length (mm)	Thickness (mm)	Sphericity
Mexico	β_0	5.2152	4.7744	2.2072	0.7938
	SE	0.4783	0.9080	0.1456	0.0736
	β_1	−0.1214	−0.1125	0.0106	0.0089
	SE	0.0530	0.1006	0.0161	0.0082
	β_2	0.0044	0.0043	0.0002	−0.0003
	SE	0.0014	0.0027	0.0004	0.0002
	R^2	97.56	93.11	98.50	75.12
	SEE	0.0435	0.0826	0.0132	0.0067
China	β_0	3.5093	4.0210	2.6065	0.8662
	SE	0.3193	0.5651	0.0924	0.0642
	β_1	0.1854	0.0768	0.0216	−0.0005
	SE	0.0351	0.0621	0.0101	0.0070
	β_2	−0.0041	−0.0010	−0.0003	0.0000
	SE	0.0009	0.0016	0.0003	0.0002
	R^2	98.36	95.81	98.20	68.47
	SEE	0.0303	0.0536	0.0088	0.0061
Sudan	β_0	6.2883	5.1899	2.7719	0.8519
	SE	0.1813	0.5351	0.5380	0.0486
	β_1	−0.1163	−0.0358	−0.0122	−0.0013
	SE	0.0201	0.0594	0.0598	0.0054
	β_2	0.0041	0.0017	0.0011	0.0001
	SE	0.0005	0.0016	0.0016	0.0001
	R^2	99.56	93.00	93.15	83.90
	SEE	0.0172	0.0509	0.0512	0.0046

SE, represents standard error of each beta value; SEE, standard error of the estimate (standard deviation of the residuals).

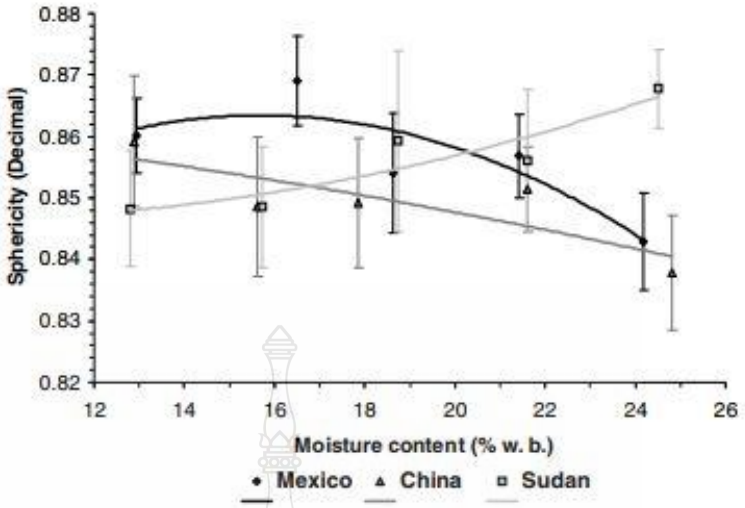


Figure 2.11 The effect of moisture content on the sphericity of Roselle seeds.

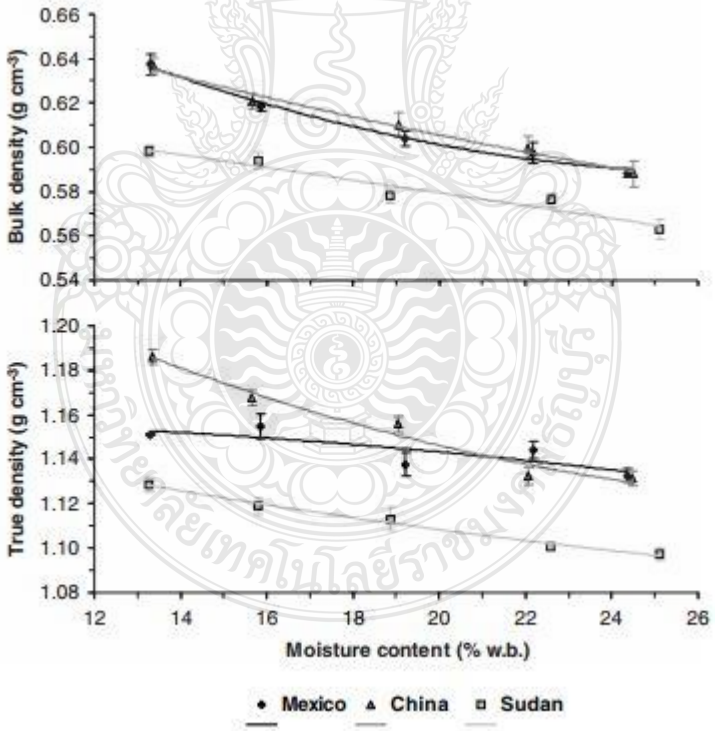


Figure 2.12 The effect of moisture content on Roselle seed bulk and true densities.

Table 2.8 The b coefficient of the quadratic model and coefficient of determining R^2 the physical properties of Roselle seed.

Cultivar	Parameter	Bulk density (kg m^{-3})	True density (kg m^{-3})	Porosity (%)	Thousand seed mass (g)
Mexico	β_0	0.7703	1.1556	33.4019	41.4283
	SE	0.0355	0.0924	4.7206	4.0486
	β_1	−0.0133	0.0006	1.1679	−2.7338
	SE	0.0039	0.0102	0.5196	0.4488
	β_2	0.0002	−0.0001	−0.0234	0.0951
	SE	0.0001	0.0003	0.0138	0.0120
	R^2	98.91	67.55	96.18	99.52
	SEE	0.0028	0.0074	0.3769	0.3684
China	β_0	0.7141	1.3176	45.8460	30.8532
	SE	0.0402	0.0676	4.5508	0.4056
	β_1	−0.0069	−0.0125	0.0010	0.2839
	SE	0.0044	0.0074	0.4991	0.0445
	β_2	0.0001	0.0002	0.0033	0.0044
	SE	0.0001	0.0002	0.0132	0.0012
	R^2	98.54	97.33	82.82	99.98
	SEE	0.0032	0.0054	0.3647	0.0385
Sudan	β_0	0.5832	1.1822	51.0441	30.9032
	SE	0.0154	0.0211	2.1789	3.5302
	β_1	0.0032	−0.0048	−0.5348	0.4076
	SE	0.0017	0.0023	0.2358	0.3921
	β_2	−0.0002	0.0001	0.0174	0.0016
	SE	0.0000	0.0001	0.0061	0.0105
	R^2	99.55	98.98	96.43	98.82
	SEE	0.0014	0.0019	0.1923	0.3357

SE, standard error of each beta value; SEE, standard error of the estimate (standard deviation of the residuals)

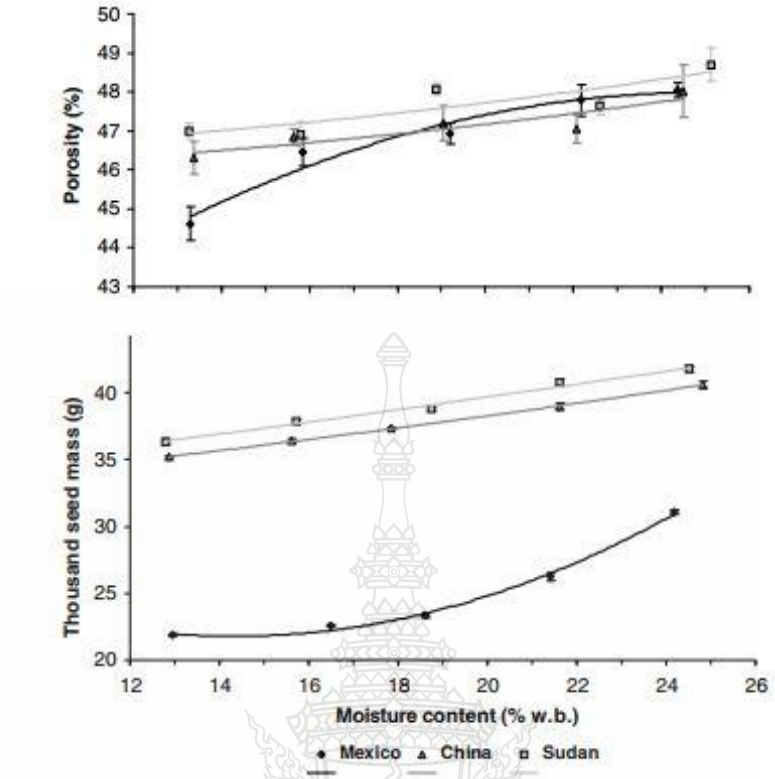


Figure 2.13 Effect of moisture content on the porosity of thousand mass Roselle seeds.

Table 2.9 Coefficients b of the quadratic model and coefficient of determination R2 for Roselle static coefficient of friction

Parameter	Concrete	Galvanised	Glass	Wood
β_0	0.2511	0.1936	0.1374	0.0870
SE	0.0271	0.0452	0.0446	0.0916
β_1	0.0075	0.0078	0.0114	0.0133
SE	0.0030	0.0050	0.0049	0.0102
β_2	−0.0002	−0.0001	−0.0002	−0.0002
SE	0.0001	0.0001	0.0001	0.0003
R^2	95.95	95.04	97.68	96.11
SEE	0.0027	0.0045	0.0045	0.0092

SE, standard error of each beta value; SEE, standard error of the estimate (standard deviation of the residuals).

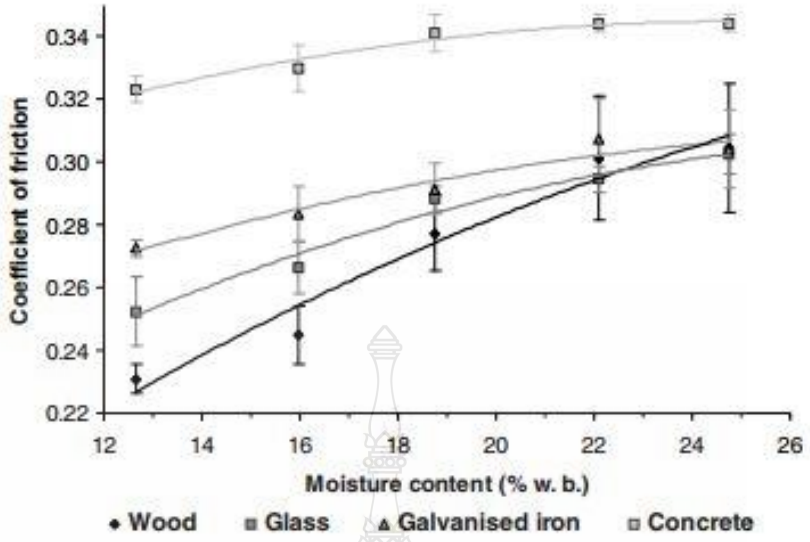


Figure 2.14 Effect of moisture content on Roselle seeds static coefficient of friction.

Efthymia Alexopoulou et al. [32] From the evaluation of the proximate dominant as a solid biofuel calorific value and the analysis of the elements, the calorific value was determined in a bomb calorimeter with moisture, volatile matter, ash content, and fixed carbon. The net calorific value (NCV) was 3852 kcal/kg and the gross calorific value (GCV) of the remaining biomass was 4035 kcal/kg. The ash content was quite high and it varied from 9.21% (Kaiima 93, 2012) to 11.75% (C864, 2012), while the nitrogen content was quite low, ranging from 0.73% (C855) to 0.94% (Kaiima 93).

Table 2.10 The percentage of oil content in the seeds and fatty acid profile (Aliartos-GR).

Hybrids/years		Oil content (%)	Oil profil					
			Ricin oleic acid (%)	Oleic acid (%)	Linoleic acid (%)	Palmitic acid (%)	Stearic acid (%)	Other fatty acids (%)
2011	Kaiima 71	48.90	84.80	3.16	4.01	1.89	2.03	4.11
	Kaiima 75	52.10	84.60	3.64	4.12	1.13	1.47	5.04
	Kaiima 93	46.60	86.87	3.08	4.12	1.51	1.46	2.96
	Mean	49.20	85.42	3.29	4.08	1.51	1.65	4.04
2012	Kaiima 93	48.80	85.36	3.12	4.09	2.08	2.09	3.26
	C-854	49.70	86.36	3.54	4.37	1.64	1.76	2.33
	C-855	48.90	86.31	3.14	4.18	1.33	1.53	3.51
	C-856	46.70	85.43	3.11	4.19	1.14	1.65	4.48
	C-864	45.80	86.28	3.2	4.17	1.77	2.01	2.57
	Mean	47.98	85.95	3.22	4.20	1.59	1.81	3.23
2014	Kaiima 93	49.50	84.00	3.74	4.73	1.57	1.77	4.19
	C-854	47.20	86.28	3.00	4.01	1.04	1.49	4.18
	C-855	46.60	85.63	3.63	4.19	1.81	1.57	3.17
	C-856	48.70	85.58	3.20	4.21	1.72	1.35	3.94
	C-1002	48.70	84.92	3.19	4.25	1.34	2.04	4.26
	C-1008	45.50	84.45	3.85	4.90	1.26	1.89	3.65
	Mean	47.70	85.14	3.44	4.38	1.46	1.69	3.90

Table 2.11 The calorific value which is; gross calorific value (GCV), net calorific value (NCV), proximate analysis (ash, fixed carbon and volatiles) and elemental analysis (CHN) for stem, leaves and entire plant harvested in Aliartos-GR in 2011 and 2012

Years	Hybrids	Calorific value		Proximate analysis			Elemental analysis		
		GCV ($MJ\,kg^{-1}$)	NCV ($MJ\,kg^{-1}$)	Ash (%)	Fixed carbon (%)	Volatiles (%)	C (%)	H (%)	N (%)
2011 Leaves	Kaiima 71	17.74	16.56	13.84	10.66	75.51	41.44	5.57	2.63
	Kaiima 75	17.43	16.27	14.07	11.74	74.20	41.24	5.51	3.52
	Kaiima 93	16.90	15.80	13.72	11.42	74.87	42.15	5.23	2.81
	Mean	17.36	16.21	13.88	11.278	74.86	41.61	5.44	2.99
Stems	Kaiima 71	17.04	15.88	9.05	13.54	77.42	41.64	5.51	0.45
	Kaiima 75	17.75	16.59	7.12	15.22	77.66	41.24	5.53	0.41
	Kaiima 93	17.30	16.16	9.09	13.17	77.75	42.09	5.43	0.80
	Mean	17.36	16.21	8.42	13.98	77.61	41.66	5.49	0.55
2012 Whole plant	Kaiima 93	16.96	15.75	9.21	13.91	76.89	42.21	5.71	0.94
	C-854	16.98	15.76	9.93	13.17	76.91	41.84	5.73	0.79
	C-855	16.97	15.77	9.43	13.98	76.60	42.74	5.70	0.73
	C-856	16.92	15.78	11.47	12.68	75.85	41.50	5.41	0.95
	C-864	16.65	15.48	11.75	12.61	75.64	41.40	5.55	0.81
	Mean	16.90	15.71	10.36	13.27	76.38	41.94	5.62	0.8

John S. Roberts et al [33] Drying from Griesling's Vitis vinifera grapes and Cabernet Franc is a waste of white and red wines, respectively, and a variety of grapes from Vitis labrusca represent waste from the Pomace grape juice production process derived from the wine and grape juice producers in New York. The seed produced were

differentiated using a separator (Turbo Fisher / Bertocchi Inc., ITALY). The Riesling seeds of about 6 mm are the smallest of three species. The seeds are separated from the waste with the help of a screen of 8 mm. When separated, they are washed to remove the remaining residue. The original moisture content of all the sample seeds is determined by convective hot air drying at temperature of 70 ºC in a vacuum oven (Model 3608, Lab-Line Instrument, Inc.) for 24 hours. The diagram of the machine used for drying is shown in Figure 2.15. The basic design of the convection dryer inside the polycarbonate air tank using the invention. The net basket balance is connected to a desktop computer by the RS-232 cable and the weight reduction of the sample is recorded online at an interval of every 30 seconds throughout the process using the Balance Link, Mettler Toledo, Inc., Hightstown, NJ. The temperature that is utilized to dry the seeds before the oil is extracted may affect the final oil quality negatively. However, very little information is known about the extent of the damage caused by the heat during drying process, before the extraction of the oil. Therefore, the temperature range used in this study is 40, 50 and 60 ºC. A low temperature range suitable for drying the seeds. From the equilibrium moisture content of various reports, it is found that the balance content of moisture in the seeds is less than the initial one, due to the variation in relative humidity during the drying. Therefore, the balanced content of moisture can be assumed to be 0 g / g dry solids. This presumption is true only at the beginning of the drying. But when the moisture has totally dried up, it will move towards the balanced moisture content, resulting in a huge impact on both the slope and straightness of the normal drying curve, as shown in Figure 2.16. Based on the moisture content equilibrium of various reports, it is found that the initial moisture content is higher than the balanced moisture content due to the variations in relative humidity during drying. Therefore, the moisture content balance can be assumed to be 0 g/g dry solids. This presumption is true only at the initial stage of drying, but when the moisture finally dries up, it will approach the balanced moisture content, resulting in a huge impact on both the slope and straightness of the normal drying curve, as can be seen in Figure 2.17, and drying curves at 40, 50, and 60º C, Riesling seeds(a), Cab Franc seeds(b), and

Concord seeds(C) as in Figure 2.18. Results of Equilibrium moisture content in Table 2.7

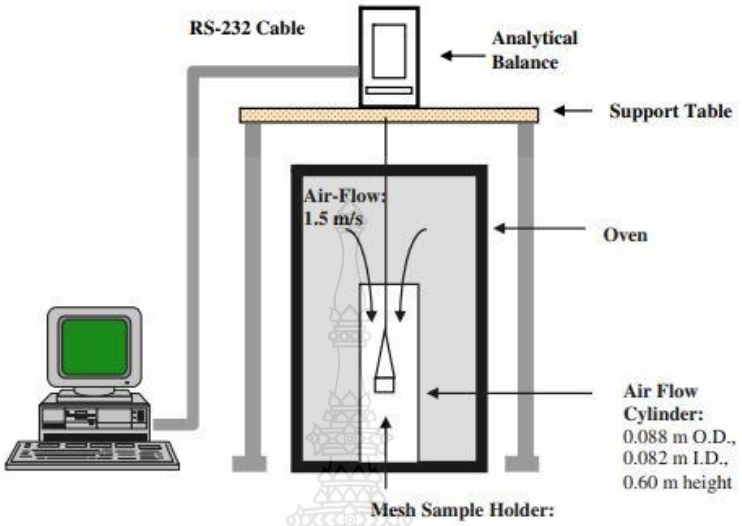


Figure 2.15 Hot-air convection drying apparatus.

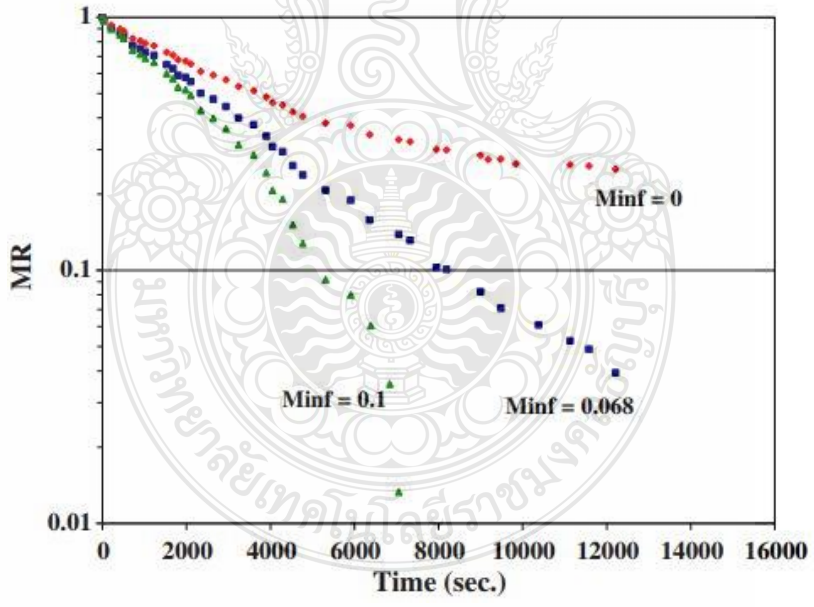


Figure 2.16 The effect of moisture content equilibrium on the normal plot of unachieved moisture content.

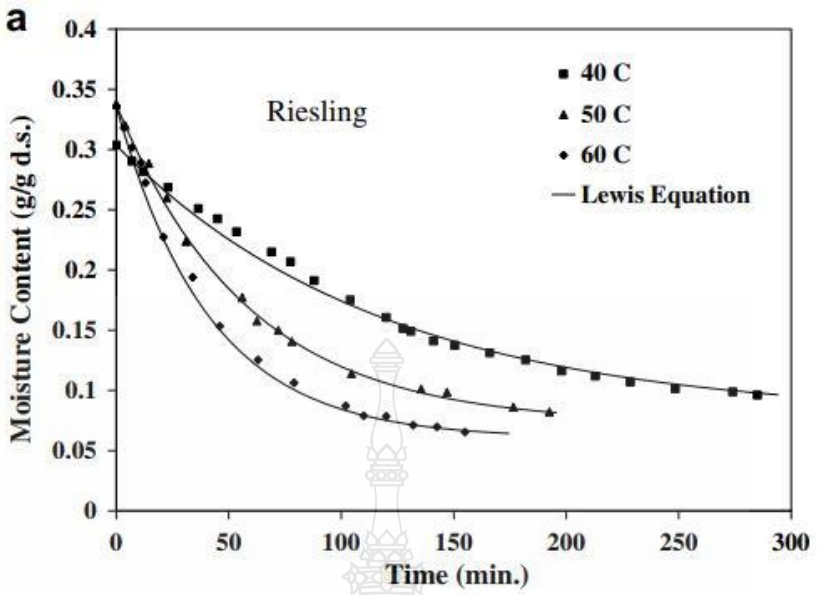
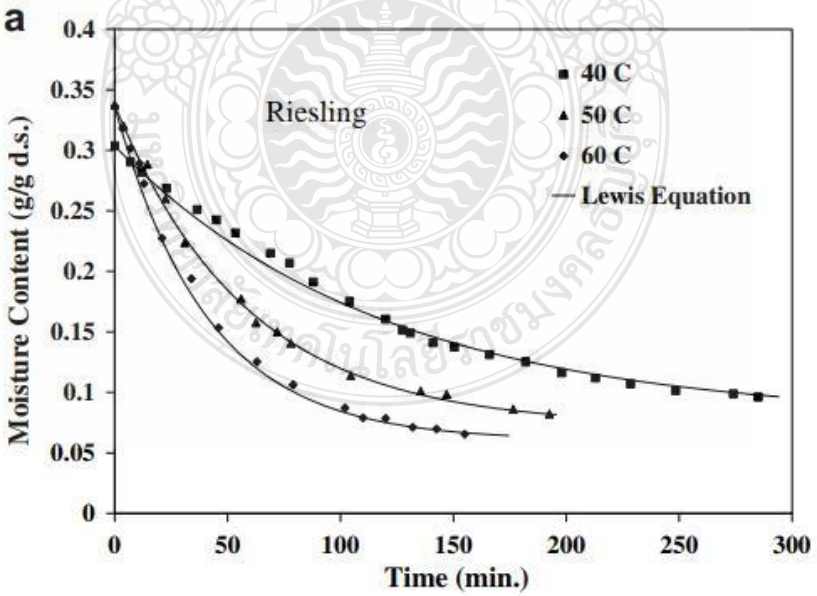


Figure 2.17 The effect moisture content equilibrium on the normal plot of unachieved moisture content.



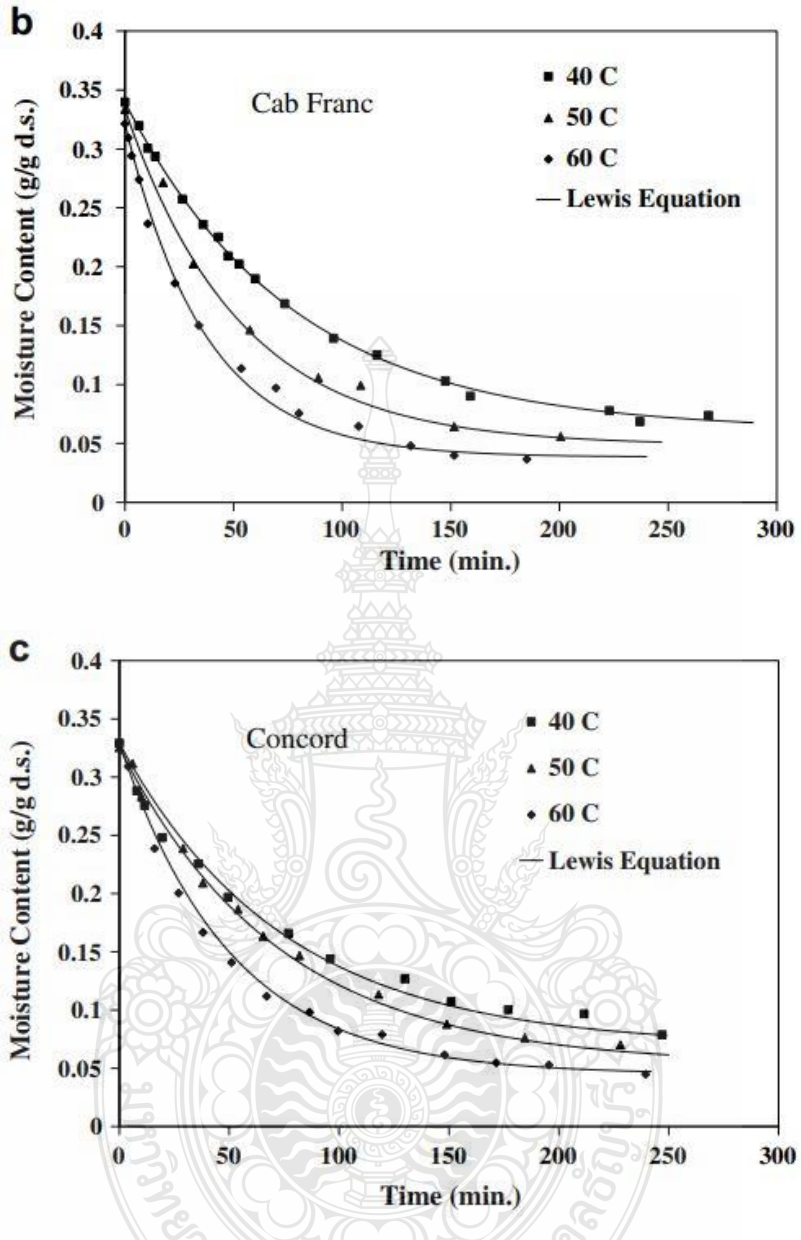


Figure 2.18 The effect of moisture content equilibrium on the normal plot of unachieved moisture content.

Table 2.12. Moisture content equilibrium.

Grape seed variety	M_i (g/g d.s.)	M_e (g/g d.s.)		
		40 °C, 12% ERH	50 °C, 7.5% ERH	60 °C, 4.5% ERH
Riesling	0.344 ± 0.024	0.102 ± 0.005	0.078 ± 0.006	0.061 ± 0.006
Cab Franc	0.387 ± 0.022	0.074 ± 0.008	0.054 ± 0.004	0.040 ± 0.001
Concord	0.324 ± 0.024	0.074 ± 0.001	0.057 ± 0.001	0.051 ± 0.006

G.P.P. Kamatou and A.M. Viljoen [34] made a comparison of fatty acid of methyl esters of palm and palmist oil as determined by GCxGC–ToF–MS and GC–MS/FID. A total number of five samples of palm nuts were obtained from Nigeria (n = 1), Ghana (n = 1), and Cameroon (n = 3) and a sample of two palmist oil (commercially available and raw oils) were obtained from Cameroon. The comprehensive chromatography gas was paired up with the time of flight mass spectrometry (GCxGC–ToF–MS) and a flame ionization detector (GC–MS/FID) and were used to ascertain the fatty acids, and their methyl esters (FAMEs) in these two products. The major compounds detected using GCxGC–ToF–MS and GC–MS/FID in palm oil were as follows: 9-octadecenoic acid methyl ester of 33.7–45.2%, hexadecanoic acid methyl ester of 27.4–44.1%, 9–12-octadecadienoic acid methyl ester of 8.5–13.4% and stearic acid methyl ester of 2.7–13.7%. Some of the FAMEs were minor and region-specific. The palmist oil with a one- and two-dimensional GC showed minimal variations as follows: hexadecenoic acid methyl ester of 11.2–18.4%, 9-octadecenoic acid methyl of 13.0–17.6%, tetra decanoic acid methyl ester of 15.1–20.6% and dodecanoic acid methyl ester of 30.4–40.1%, being the most prominent. Identified FAMEs such as (Z, Z, Z) - 9,12,15-octadecadienoic acid methyl ester; tetra decanoic acid, 12-methyl, methyl ester were used to differentiate between the two palmist oils when using the two dimensional GC. Generally, fewer FAMEs could be detected by GC–MS/FID, usually less than 10. When the oils were separated using two dimensional GC, this number doubled.

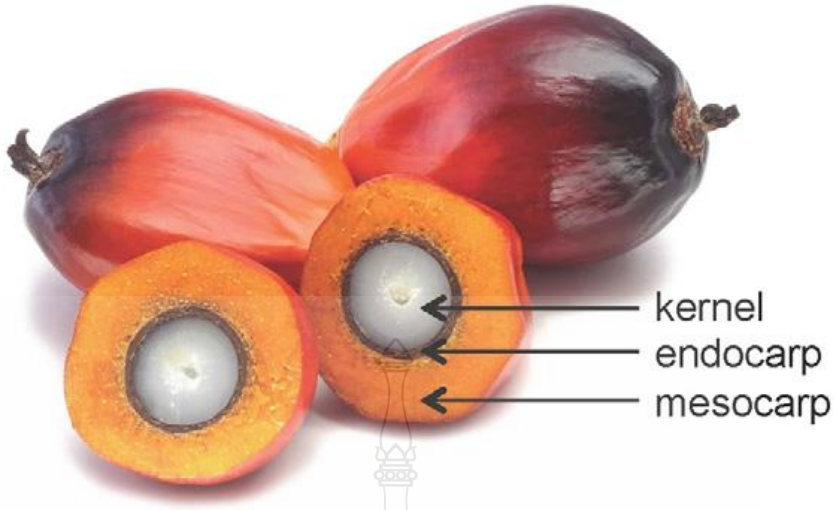


Figure 2.19 The source of palm nut (mesocarp) and palmist oil (kernel).

Table 2.13 Fatty acid methyl esters (% area) in palm nuts from three different countries.

Peak number	Compounds	RT (GC X GC)		CAPOC1		CAPOC2		RPOC		CAPOG		CAPON	
		1st Dim (s)	2nd Dim (s)	GC-MS/FID	2D	GC-MS/FID	2D	GC-MS/FID	2D	GC-MS/FID	2D	GC-MS/FID	2D
1	Decanoic acid, methyl ester	598	0.740	-		-	-		-	-	2.0	-	-
2	Tridecanoic acid, methyl ester	878	0.765				tr		0.1		1.7		3.0
3	Tetradecanoic acid, methyl ester	942	0.775	0.8	0.6	0.5	0.7	1.0	1.1	1.0	1.8		5.0
4	Hexadecanoic acid, methyl ester	1002	0.785	44.1	32.3	37.6	30.5	43.0	37.1	45.0	27.4	41.0	35.7
5	Hexadecanoic acid, 15- methyl ester	1060	0.825	-	1.5		tr	-	0.5	-	-	-	0.1
6	Stearic acid, methyl ester	1114	0.815	4.8	2.7	5.2	6.0	4.1	5.8	4.3	13.7	5.9	7.9
7	(Z)-9-Octadecenoic acid, methyl ester	1118	0.790	37.9	40.9	45.2	43.0	40.1	38.5	37.2	33.7	34.4	33.8
8	(Z,Z)-9,12-Octadecadienoic acid, methyl ester	1136	0.750	10.4	8.5	10.2	9.7	10.2	9.0	12.0	13.4	10.7	11.3
9	(Z,Z,Z)-9,12,15-Octadecadienoic acid, methyl ester	1162	0.725	-	-	-	0.7		-	-	0.6	-	0.6
10	Tetradecanoic acid, 12-methyl, methyl ester	1200	1.400	-	2.1	-	-		0.3	-	0.2	-	0.1
11	Oxiraneundecanoic acid, 3-pentyl, methyl ester (cis) *	1200	1.480	-	1.7	-	-	-	-	-	0.6	-	-
12	Eicosanoic acid, methyl ester	1220	0.830	0.4	tr	0.2	0.6	0.5	0.2	0.4	0.2	0.3	0.1
13	Nonanoic acid, methyl ester*	1122	0.845	-	-	-	-		-	-	0.3	-	0.7
14	2-Pentanoic acid-2-methoxy-3-methyl-methyl ester *	1236	1.330	-	1.3	-	-		-	-	-	-	-
15	Carbamic acid, methyl ester	1248	1.020	-			-		0.3	-	-	-	-
16	Hexanoic acid, 3-ethyl-methyl ester	1286	1.190						1.8				
17	6-Octadecenoic acid, methyl ester*	1292	0.851		0.4		tr	-	0.5	-	-		-
18	2,6-Dimethyl-8-oxooct-2,6-dienoic acid, methyl ester *	1294	0.720	-	1.7		2.3	-	-	-	-	-	-
19	11,14-Eicosadienoic acid, methyl ester	1298	0.855	-	0.4	-	-	-	-	-	-	-	-
20	Butanedioic acid, methoxy-, dimethyl ester *	1306	1.540	-	0.8	-	-	-	-	-	-	-	-
21	Unknown 1	1310	0.715	-	1.2	-	1.6	-	0.1	-	0.5	-	0.1
22	Octadecanoic acid, 3-oxo-methyl ester *	1316	0.710	-	0.8	-	-	-	0.3	-	-	-	-
23	Pentadecanoic acid, 3-oxo-methyl ester *	1320	0.680	-	-	-	0.3	-	-	-	-	-	0.5
24	Unknown 2	1336	1.155	-	-	-	-	-	3.9	-	-	-	-
25	Unknown 3	1354	1.080	-	2.9	-	-	-	-	-	-	-	-
26	Unknown 4	1362	1.145		-	-	-		-	-	3.2	-	-
27	Tetradecanoic acid, 2-oxo-, methyl ester *	1387	1.096	-	-	-	-	-	0.4	-	-	-	-
28	Heptadecanoic acid, 3-oxo-methyl ester *	1423	0.840	-	-	-	0.3	-	-	-	-	-	-
Total saturated fatty acid methyl esters				**50.1**	**35.6**	**43.5**	**37.38**	**48.6**	**44.3**	**50.7**	**47.1**	**47.2**	**52.4**
Total unsaturated fatty acid methyl esters				**48.3**	**64.2**	**55.4**	**57.9**	**50.3**	**55.6**	**49.2**	**52.2**	**45.1**	**46.5**

RT represents the retention time in seconds, provisionally identified by NIST and Adams libraries. TR represents the trace amount (b0.1%); first dimensional GC (1st Dim), second dimensional GC (2nd Dim). CAPOC1 refers to palm oil commercially available from Cameroon; CAPOC2 refers to palm oil commercially available from Cameroon. RPOC stands for raw palm oil from Cameroon. CAPOG refers to palm oil commercially available from Ghana. CAPON represents refined palm oil from Nigeria. Unknown 1–4: Unknown FAMEs (similarity b750). To calculate the total unsaturated fatty acids = polyunsaturated fatty acids + monounsaturated fatty acids. The sum total of unsaturated fatty acids + saturated fatty acids will not sum up to 100% because each item has a small amount of other fatty substances that are neither saturated nor unsaturated.

Table 2.14 Fatty acid methyl esters (% area) in palmist oil obtained by two different processing methods.

Peak number	Compounds	RT (GC X GC)		CAPMO		RPMO	
		1st Dim (s)	2nd Dim (s)	GC–MS/FID	2D	GC–MS/FID	2D
1	Hexanoic acid, methyl ester	260	0.650	-	-	-	tr
2	Octanoic acid, methyl ester	434	0.710	-	1.4	-	3.2
3	Decanoic acid, methyl ester	598	0.740	-	4.6	-	3.6
4	Tridecanoic acid, methyl ester	878	0.765	-	tr	-	0.1
5	Tetradecanoic acid, methyl ester	942	0.775	20.6	15.1	19.5	17.8
6	Hexadecanoic acid, methyl ester	1002	0.785	18.4	14.1	18.4	11.2
7	Hexadecanoic acid, 15-methyl, methyl ester*	1060	0.825	-	tr	-	0.2
8	Dodecanoic acid, methyl ester	1106	0.505	30.4	35.2	36.1	40.1
9	Stearic acid, methyl ester	1114	0.815	6.5	4.5	6.4	5.0
10	(Z)-9-Octadecenoic acid, methyl ester	1118	0.790	13.8	17.6	13.0	14.0
11	(Z,Z)-9,12-Octadecadienoic acid, methyl ester	1136	0.750	3.9	2.9	5.2	4.1
12	Nonanoic acid, methyl ester*	1141	0.770	-	tr	-	0.2
13	(Z,Z,Z)-9,12,15-Octadecadienoic acid,methyl ester	1162	0.725	-	0.2	-	
14	Tetradecanoic acid, 12-methyl, methyl ester	1200	1.400	-	2.2	-	-
15	Eicosanoic acid, methyl ester	1220	0.875	0.4	0.7	0.5	0.2
16	Unknown 1	1294	0.720	-	0.3	-	0.2
17	Unknown 5	1316	0.710	-	0.5	-	-
18	Unknown 4	1398	0.458	-	0.1	-	-
Total saturated fatty acid methyl esters				**76.3**	**75.6**	**80.9**	**81.4**
Total unsaturated fatty acid methyl esters				**17.7**	**23.8**	**18.2**	**18.5**

RT represents the retention time in seconds, provisionally identified by NIST and Adams libraries. TR being the trace amount (< 0.1%); first dimensional GC (1st Dim), second dimensional GC (2nd Dim); palm oil commercially available from Cameroon

(CAPOC1); palm oil commercially available from Cameroon (CAPOC2); raw palm oil from Cameroon RPOC); palm oil commercially available from Ghana (CAPOG), refined palm oil from Nigeria (CAPON); Unknown 1–4: Unknown FAMEs (similarity < 750). Total calculate the unsaturated fatty acids = polyunsaturated fatty acids + monounsaturated fatty acids. The sum of total unsaturated fatty acids + saturated fatty acids will not add to 100% because each item has a small amount of other fatty substances that are neither saturated nor unsaturated.

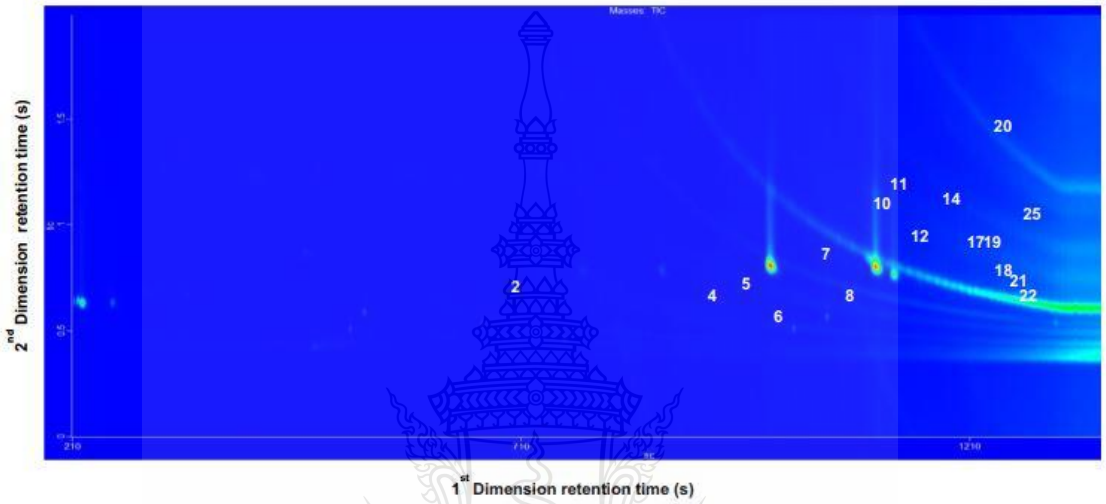


Figure 2.20 Second dimension contour plot of palm oil from Cameroon. For corresponding FAME, refer to the peak number in Table 2.13

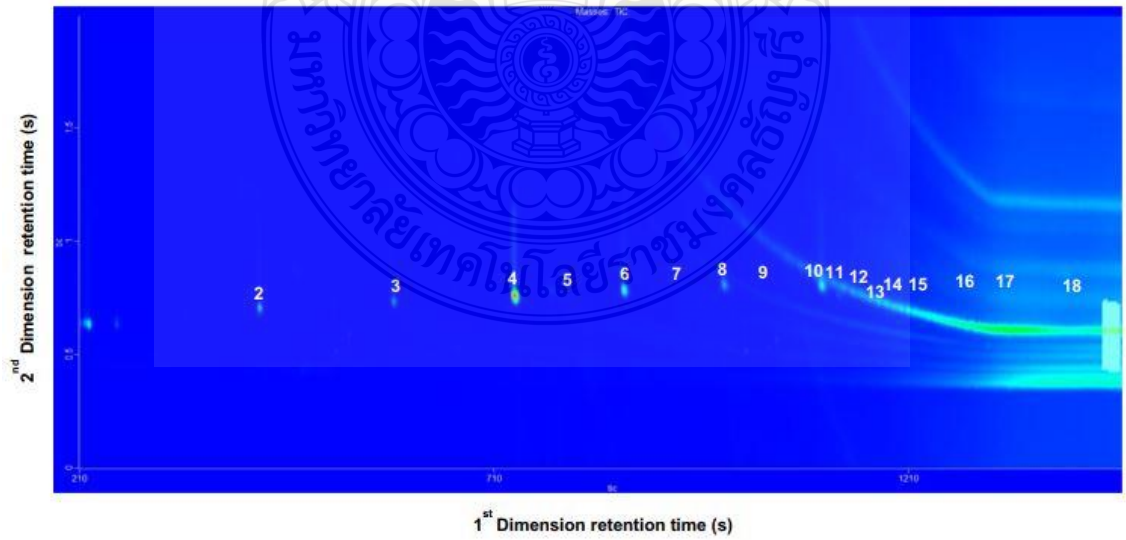


Figure 2.21 Second dimension contour plot of palmist oil. For corresponding FAME, refer to the peak number in Table 2.14

Mohammed Danish and Maniruddin Nizami [35] made a study on the complete data analysis of the fatty acids of flaxseed oil utilizing the GC-FID method chromatography (GC) with a flame ionization detector (FID). It was found that the flaxseed oil can change into fatty acid methyl ester (FAME). The GC was analyzed with FID and the retention time of the different fatty acid present in the flaxseed oil was observed. The time retained after observation was compared with the standard fatty acid to confirm the actual fatty acid present in the flaxseed oil. Part of the data is used in the article titled "The process of variable optimization for biodiesel production by transesterification of flaxseed oil and the characteristics of biodiesel produced." The data indicates that the fatty acid as shown in Table 1 represent the Supelco 37 standard FAME data for the purpose of comparison. Table 2 shows the GC-FID data for flaxseed oil when changing FAME. The chromatogram of Supelco 37 standards is shown in Fig. 2.20 and flaxseed oil converted FAME chromatogram is shown in Fig. 2.21.

Table 2.15 Supelco 37 component Mix FAME data analysis used for calibration, quantitation & identification of the unknown peaks in the oil samples.

SN	Fatty acids in CRM	Fatty acid groups	RT	Short name	% Area
1	C4:0—Butyric acid	SAFA	6.519	C4:0	1.69614
2	C6:0—Caproic acid	SAFA	6.913	C6:0	3.13618
3	C8:0—Caprylic acid	SAFA	7.606	C8:0	3.22269
4	C10:0—Capric acid	SAFA	8.706	C10:0	3.3805
5	C11:0—Undecanoic acid	SAFA	9.424	C11:0	1.84709
6	C12:0—Lauric acid	SAFA	10.264	C12:0	3.94123
7	C13:0—Triundecanoic acid	SAFA	11.242	C13:0	2.06048
8	C14:0—Myristic acid	SAFA	12.394	C14:0	4.32965
9	C14:1—Myristoleic acid	MUFA	13.479	C14:1	2.13117
10	C15:0—Pentadecanoic acid	SAFA	13.771	C15:0	2.23474
11	C15:1—*cis*-10-Pentadecenoic acid	MUFA	15.103	C15:1	2.15963
12	C16:0—Palmitic acid	SAFA	15.456	C16:0	6.3978
13	C16:1—Palmitoleic acid	MUFA	16.810	C16:1	2.24513
14	C17:0—Heptadecanoic acid	SAFA	17.488	C17:0	1.56535

SN	Fatty acids in CRM	Fatty acid groups	RT	Short name	% Area
15	C17:1—*cis*-Heptadecenoic acid	MUFA	18.882	C17:1	2.21837
16	C18:0—Stearic acid	SAFA	19.553	C18:0	4.69103
17	C18:1—*trans*-9-Elaidic acid	TFA	20.348	C18:1n9t	2.28174
18	C18:1 (n-9)—Oleic acid	MUFA/ω9FA	20.723	C18:1n9c	4.5848
19	C18:2—*trans*-Linolelaidic acid	TFA	21.617	C18:2n6t	2.10519
20	C18:2 (n-6)—Linoleic acid	PUFA	22.423	C18:2n6c	2.09427
21	C20:0—Arachidic acid	SAFA	23.401	C20:0	4.68651
22	C18:3 (n-6)—g-Linolenic acid	PUFA/ω6FA	23.601	C18:3n6	1.91131
23	C18:3 (n-3)—a-Linolenic acid (ALA)	PUFA/ω3FA	24.314	C18:3n3	1.84683
24	C20:1 (n-9)—*cis*-11-Eicosenic acid	MUFA	24.458	C20:1	2.3618
25	C21:0—Heneicosanoic acid	SAFA	25.262	C21:0	2.36044
26	C20:2—*cis*-11,14-Eicosadienoic acid	PUFA	26.203	C20:2	2.07894
27	C22:0—Behenic acid	SAFA	27.281	C22:0	4.67455
28	C22:3n6—*cis*-8,11,14-Eicostrienoic acid	PUFA/ω6FA	27.548	C20:3n6	1.69522
29	C20:3n3—*cis*-11,14,17-Eicosatrienoic acid	PUFA/ω3FA	28.407	C20:3n3	1.38465
30	C22:1 (n-9)—Erucic acid	MUFA/ω9FA	28.553	C22:1n9	2.32792
31	C20:4 (n-6)—Arachidonic acid	PUFA/ω6FA	28.591	C20:4n6	1.74384
32	C23:0—Tricosanoic acid	SAFA	29.504	C23:0	2.46567
33	C22:2—*cis*-13,16-Docasadienoic acid	PUFA	30.705	C22:2	2.114
34	C20:5 (n-3)—*cis*-5,8,11,14,17-Eicosapentaenoic acid (EPA)	PUFA/ω3FA	31.251	C20:5n3	1.53761
35	C24:0—Lignoceric acid	SAFA	32.030	C24:0	4.75565
36	C24:1—Nervonic acid	MUFA	33.684	C24:1	2.14213
37	C22:6 (n-3)—*cis*-4,7,10,13,16,19-Docosahexaenoic acid (DHA)	PUFA	37.833	C22:6n3	1.58971

Table 2.16 The relative percentage of fatty acids in the total fat of the flaxseed oil.

SN	Fatty acids in flaxseed oil	Fatty acid groups	RT	Peak area (FAME)	% Fat (of total fat)
1	C14:0—Myristic acid	SAFA	12.388	0.569951	0.046
2	C16:0—Palmitic acid	SAFA	15.457	70.39929	5.687
3	C16:1—Palmitoleic acid	MUFA	16.806	1.19302	0.096
4	C18:0—Stearic acid	SAFA	19.571	68.69044	5.578
5	C18:1—*trans*-9-Elaidic acid	TFA	20.441	1.06464	0.086
6	C18:1 (n-9)—Oleic acid	MUFA/ω9FA	20.757	253.6431	20.591
7	C18:2—*trans*-Linolelaidic acid	TFA	21.649	1.10871	0.09
8	C18:2 (n-6)—Linoleic acid	PUFA/ω6FA	22.445	194.69879	15.801
9	C20:0—Arachidic acid	SAFA	23.412	2.50581	0.204
10	C18:3 (n-6)—g-Linolenic acid	PUFA	23.705	2.87935	0.234
11	C18:3 (n-3)—a-Linolenic acid (ALA)	PUFA/ω3FA	24.37	633.32971	51.376
12	C22:0—Behenic acid	SAFA	27.275	2.17424	0.178
13	C24:0—Lignoceric acid	SAFA	32.014	0.394852	0.034
14	Sum of Omega-3 (n-3)	ω3FA	—	—	51.376
15	Sum of Omega-6 (n-6)	ω6FA	—	—	15.801
16	Sum of Omega-9 (n-9)	ω9FA	—	—	20.591
17	Saturated fats (SAFA)	SAFA	—	—	11.727
18	Trans-fats (TFA)	TFA	—	—	0.176
19	Monounsaturated fats (MUFA)	MUFA	—	—	20.687
20	Polyunsaturated fats (PUFA)	PUFA	—	—	67.41
21	Total Unsaturated fats (TUFA)	TUFA	—	—	88.097

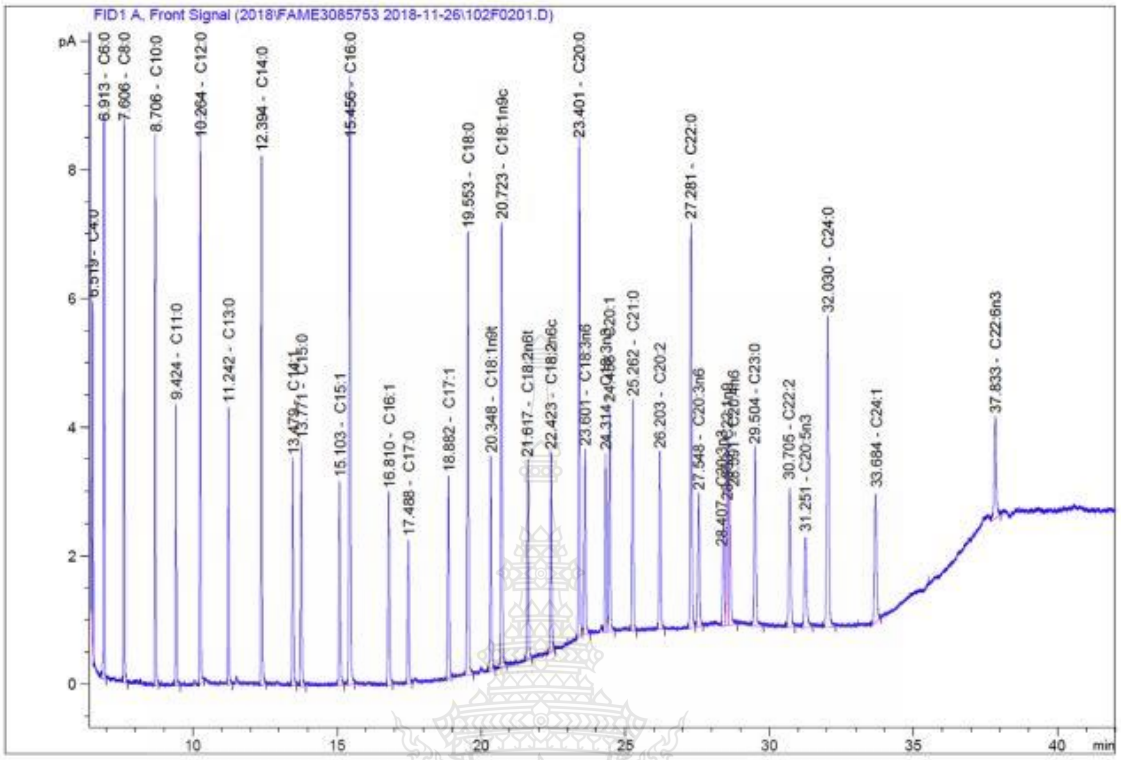


Figure 2.22 GC-FID chromatogram of supelco 37 Mix FAME component,

Cat: CRM47885, Lot: XA19807V.

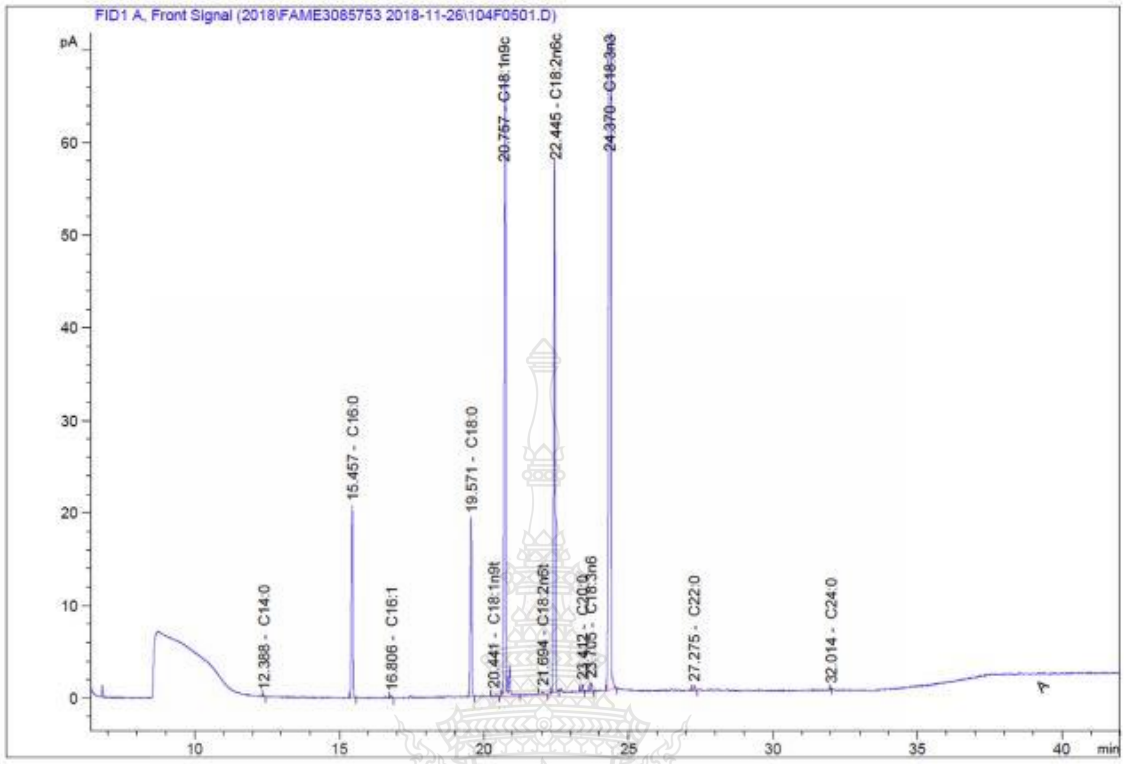


Figure 2.23 GC-FID Chromatogram of flaxseed oil with peak label.

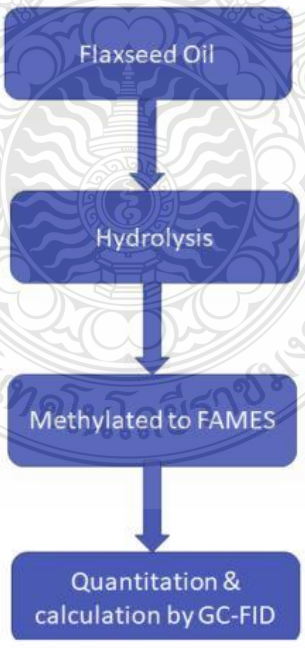


Figure 2.24 Flow chart of the fatty acid analysis of flaxseed oil.

Evandro Pereira et al. [36] studied about the development and validation of analytical methodology by GC-FID. It was developed to determine the content of biodiesel in fatty acid methyl ester (FAME). Biodiesel has internal standards (IS) which include: methyl heptadecanoate (MHD), and non-decanoate (MND), respectively. These internal standards can also be found in bovine tallow methyl esters (BTME), but they have not been effectual. This research proposes an alternative BTME to improve the method by using hexadecyl propanoate (HDP) as an IS. As a result, a subsidiary analytical methodology using Gas Chromatography-Flame Ionization Detector (GC-FID) was developed and implemented, where HDP selected retention time between the peaks is C16:1 and C18:0 for coconut, soybeans and methyl esters BTME, with a resolution > 1.5 for the BTME in split mode of 30:1. The validity of determining the BTME content using the HDP as an IS was equal to the confidence interval of 95% for statistical test of the null hypothesis, even when only 20% of the HDP was utilized in comparing the IS concentrations as defined by EN 14103:2003 and EN 14103:2011. This enabled the analysis of the biodiesel to be performed more than five times with 1 g of HDP. Furthermore, this method enabled us to reduce the analysis time by 21.6%, without discrimination to the integration of the peaks (C6:0 to C24:1). With regards to the repeated and intermediate precision tests, the results of RSD (%) ≤ 2% was reached. Moreover, this method has proven to be strong. The HDP is a connection of fatty alcohol ester not present from feedstocks used in the breakdown of biodiesel. It brings out all of the characteristics for a good IS ideal for application through the internal standardization method as approved by EN 14103.

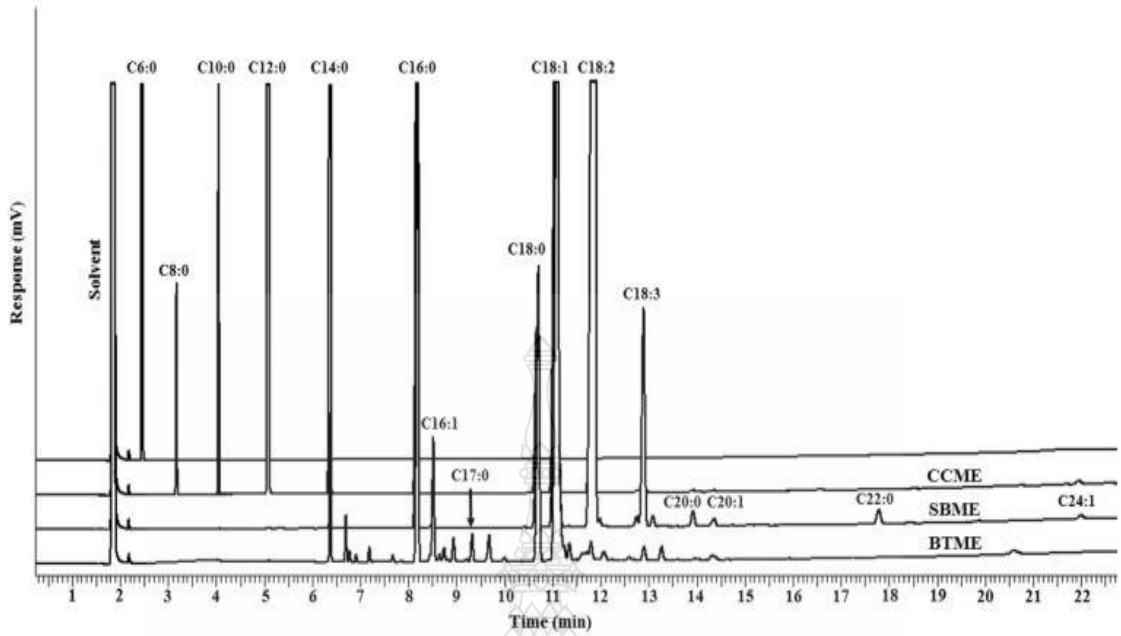


Figure 2.25 The overlapping of the chromatographic profile between the BTME, SBME, CCME and C6:0 (methyl hexanoate).

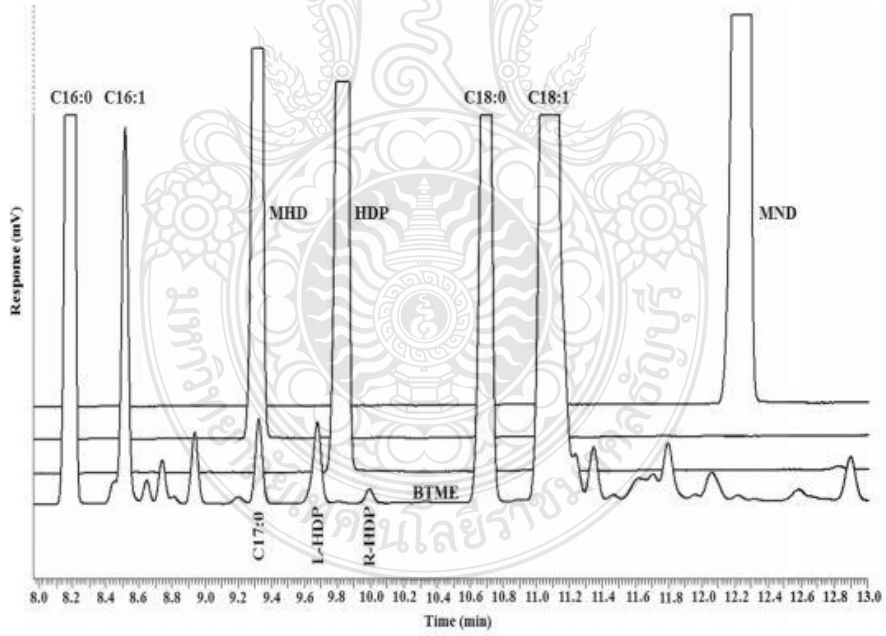


Figure 2.26 The overlapping chromatograms of the main esters in the range of t_R: 8 to 13 min out of a total of 22.91 min: the peaks of C16:0, C16:1, C17:0, L-HDP, R-HDP, C18:0

and C18:1 of BTME (blank), methyl heptadecanoate (MHD), hexadecyl propanoate (HDP) and methyl non-decanoate (MND).

Table 2.17 Determining the retention time (t_R), peak area (A), peak height (h), resolution and tailing factor (T_f) resulting in relation to the HDP and its two adjacent peaks (L-HDP and R-HDP) of BTME for split mode of 20:1, 30:1 and 50:1.

Peak	Split	t_R min	RSD %	A [µV·s]	RSD %	h [µV]	RSD %	T_f	RSD %	[a] L-HDP:HDP	[b] R-HDP:HDP
L-HDP		9.67	0.05	2878	0.81	803	0.76	0.88	0.62	1.94	1.39
HDP	20:1	9.86	0.05	54,242	0.78	14,648	1.05	0.73	0.62		
R-HDP		10.00	0.05	514	1.36	160	1.07	0.91	1.02		
L-HDP		9.68	0.03	1973	1.23	557	1.08	0.88	0.75	1.87	1.51
HDP	30:1	9.89	0.03	39,567	0.99	11,169	0.96	0.77	0.86		
R-HDP		10.00	0.03	350	1.54	108	1.10	0.92	2.04		
L-HDP		9.67	0.09	1172	0.98	337	0.86	0.89	0.91	1.82	1.67
HDP	50:1	9.84	0.09	23,335	1.41	6882	1.44	0.84	0.80		
R-HDP		9.99	0.09	201	4.93	63	2.18	0.97	2.90		

Replicate: n = 9.

[a] L-HDP: peak on the HDP left.

[b] R-HDP: peak on the HDP right.

Table 2.18 Comparing the BTME content results, using HDP and MHD to evaluate the trueness through statistical analysis by Fisher's and Student's t-tests.

Statistical analysis									
Standard	Mean (%)[(a)]	*SD*	s^2	RSD (%)	$F_{exp.}$	F_{CVF}	S_a	$t_{exp.}$	t_{CVS}
MHD	97.18	0.82	0.67	0.84	1.22	4.284	0.75	1.37	2.178
HDP	97.73	0.91	0.82	0.93					

t_{cvs}: critical values of student's t - distribution with 12 degrees of freedom.

F_{CVT}: critical values of F for the $p \leq 0.05$ with 6 degree of freedom.

RSD (%) represents the percentage of relative standard deviation.

Sa represents the aggregated standard deviation.

n = 7 number of standard.

SD is the standard deviation.

F_{exp} is the F experiment.

t_{exp} is the t experiment.

s^2 is the variance.

[a] 95% confidence level.

Table 2.19 The repeated analysis of samples with the MDH and HDP in the determination of the BTME content.

Samples BTME content									
Standard	1	2	3	4	5	6	7	Mean (%)	RSD (%)
HDP	96.0	97.4	97.8	98.1	97.6	98.2	99.0	97.7 ± 0.9	0.93
MHD	98.4	96.2	97.7	97.8	96.6	96.4	97.2	97.2 ± 0.8	0.84

n = 3 for each sample. RSD (%): percentage of relative standard deviation. Mean is at 95% confidence level.

Table 2.20 Evaluation of the intermediate precision by different analysis with the HDP in determining the BTME content.

Intermediate precision			
Analyst	BTME content (%)	Mean (%)	RSD (%)
A	98.65	99.12	1.30
B	98.13		
C	100.58		

RSD (%): percentage of relative standard deviation.

Table 2.21 Effects of the analytical parameters in determining the BTME content to evaluate the strength of the chromatographic method by Youden's test.

Analytical parameter (X/x)		BTME content (%)		$D_i^{(a-b)}$ (%)	SD_i (%)
		MCL/4[(a)]	MLCL/4[(b)]		
A/a	Injector temperature (°C)	97.97	97.98	−0.02	0.58
B/b	Detector temperature (°C)	98.16	97.79	0.36	
C/c	Oven initial temperature (°C)	98.07	97.88	0.20	
D/d	Heating rate of second ramp ($°C\,min^{-1}$)	97.65	98.30	−0.65	
E/e	Gas flow-rate ($mL\,mL^{-1}$)	98.17	97.78	0.39	
F/f	Split mode	97.91	98.04	−0.13	
G/g	Injected volume (µL)	98.30	97.65	0.65	

$Di^{(a-b)}$ - difference between the BTME contents due to small variations in the analytical parameters (%).

SDi - differences in standard deviation (%).

[a] MCL/4 - mean of four values corresponding to upper case letters (%).

[b] MLCL/4 - mean of four values corresponding to lower case letters (%).

Jaime Armendáriz et al. [37], evaluated the productivity of oil and the quality of biodiesel fuel produced from 11 forest genotype samples collected from different areas of Mexico. Oil production from seeds is evaluated on an annual basis in an experimental field at the farm level and produces biodiesel using oil conversion at the laboratory level. Quality assessments are made in accordance with the present standards, and the results showed that there was not only a variation in seed production of between 937.1kg/rai and 56.3kg/rai but in oil content as well of between 42% w/w and 54% w/w and oil production varied between 431.7kg/rai and 27 kg/ha. This results

proposed that there is a possibility of producing castor oil in the northeastern region of Mexico. The average biodiesel yield that can be produced from previously extracted oil is 1:0.84. There is a variation of between 89.16% and 84.7% in the ricolinic content of acid methyl ester. Due to its kinematic viscosity, density, water content and high CFPP, pure castor oil does not meet the standards. The results however reveal that castor oil has a potential to be mixed with diesel.

Table 2.22 First, second and third harvest of total seed produced, oil content and total production of oil, from the eleven genotype samples of castor studied.

Genotype	Harvest 1(kg/ha)	Harvest 2(kg/ha)	Harvest 3(kg/ha)	Total production of seed (kg/ha)	Oil content w/w	Total production of oil (kg/ha)
NL1M3	380.4	52.8	0	433.2^{d}	0.42^{b}	181.9
NL2M1	50.6	301.8	0	352.4^{e}	0.45^{ab}	158.6
NL2M4	93.3	368.3	0	461.6^{d}	0.47^{ab}	216.9
NL2M6	293.8	260.1	0	553.8^{c}	0.475^{ab}	263.1
NL2M7	98.8	0	0	98.8^{g}	0.45^{ab}	44.4
Chi02	20.3	207.4	709.4	937.1^{a}	0.42^{b}	393.6
Edomex	56.3	0	0	56.3^{h}	0.48^{ab}	27.0
SLP02	77.1	257.8	0	334.8^{e}	0.545^{a}	162.4
SLP05	215.3	0	0	215.3^{f}	0.505^{ab}	104.4
SLP10	632.7	0	0	632.7^{b}	0.485^{ab}	287.7
SLP11	479.5	38.8	0	518.3^{c}	0.445^{b}	251.4

The samples of the genotype in one column, followed by the same letter did not reveal any significant differences ($\leq 0.01\%$).

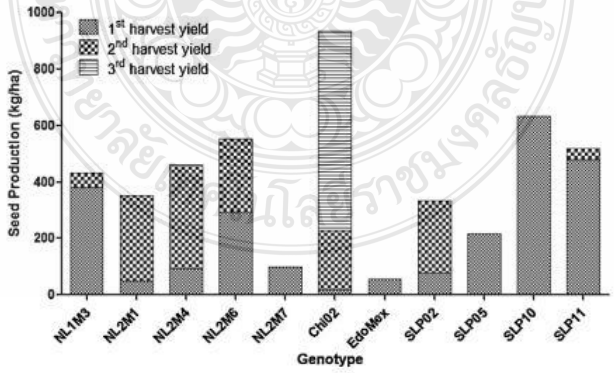


Figure 2.27 Distribution of seed production in relation to the eleven genotype samples of castor studied.

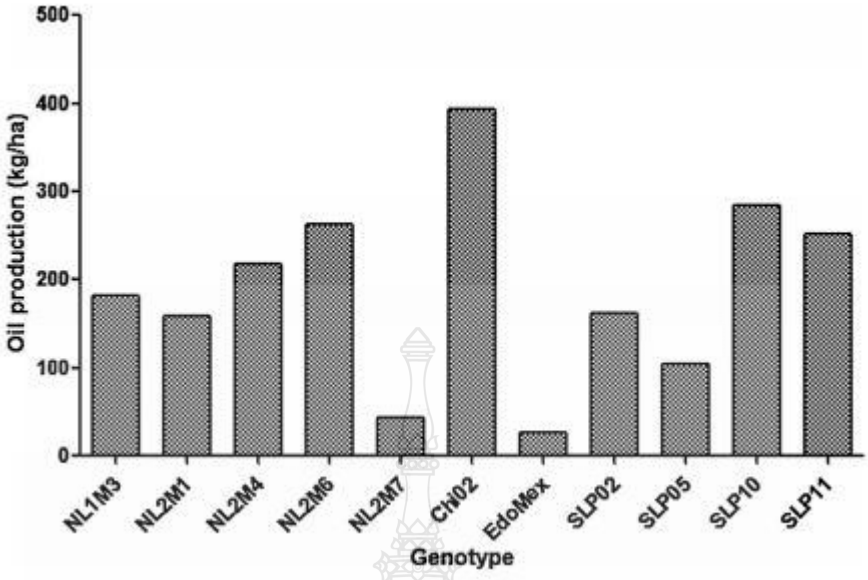


Figure 2.28 The distribution of oil production per hectare for the eleven genotype samples of castor.

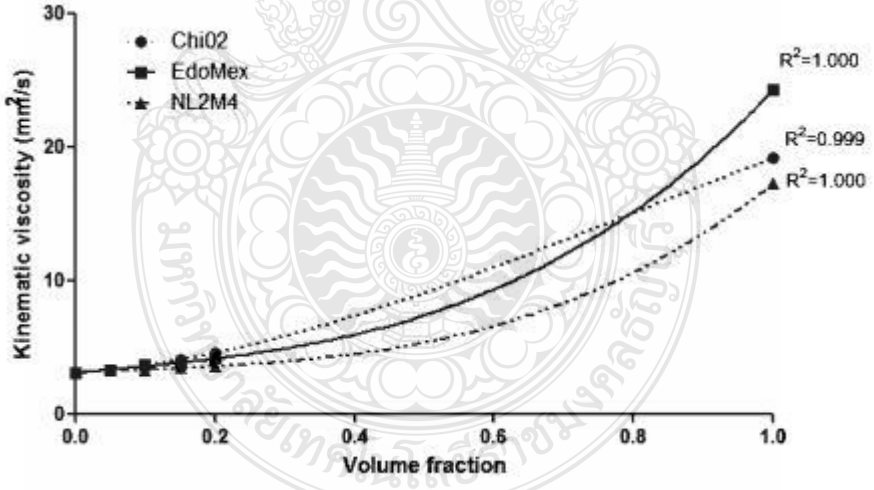


Figure 2.29 Kinematic viscosity of the biodiesel blends from three selected castor genotype samples.

Table 2.23 The quantity of oil, alcohol (methanol) and catalytic converter (NaOH) used for biodiesel production from the genotype samples studied.

Genotype	Amount of Oil (g)	Amount of alcohol (ml)	Quantity of NaOH (g)	Biodiesel (g)	Glycerin (g)	Conversion efficiency (%)
NL1M3	100	25	0.5	85.6	16	85.6
NL2M1	100	25	0.5	86.9	13.1	86.9
NL2M4	100	25	0.5	82.1	8.3	82.1
NL2M6	137.8	34.45	0.7	110.8	16.6	80.4
NL2M7	110.4	27.6	0.5	90.3	25.1	81.8
Chi02	236.5	59.12	1.2	197.3	28.9	83.4
Edomex	257.94	64.3	1.3	222.5	21.3	86.3
SLP02	100	25	0.5	93.2	8.8	93.2
SLP05	100	25	0.5	68.9	6.7	68.9
SLP11	100	25	0.5	88.1	13.6	88.1
SLP10	160	40	0.8	131.5	26.2	82.2

Table 2.24 An analysis of the profile of castor fatty acid samples of biodiesel(% w/w).

	Methyl esters of fatty acids							
Genotype	C16:0	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C18:1, 12-OH
NL1M3	0.98^{ab}	1.10^{def}	3.39^{cd}	5.16^{de}	0.62^{ab}	0.15^{a}	0.60^{a}	86.19^{ab}
NL2M1	1.03^{ab}	1.12^{ef}	3.84^{f}	5.32^{e}	0.27^{a}	0.00^{a}	0.33^{a}	86.65^{ab}
NL2M4	0.84^{a}	1.06^{bcde}	3.41^{cd}	4.50^{ab}	0.50^{ab}	0.39^{a}	0.23^{a}	87.74^{ab}
NL2M6	0.98^{ab}	0.99^{bcd}	3.99^{f}	5.15^{de}	0.54^{ab}	0.66^{a}	0.00^{a}	86.31^{ab}
NL2M7	1.16^{b}	0.96^{bc}	2.93^{a}	4.84^{bcde}	0.78^{b}	0.45^{a}	0.00^{a}	84.70^{g}
Chi02	1.12^{b}	1.21^{f}	3.72^{ef}	5.29^{de}	0.58^{ab}	0.00^{a}	0.63^{a}	86.68^{ab}
Edomex	0.81^{a}	0.94^{b}	3.08^{ab}	4.01^{a}	0.55^{ab}	0.00^{a}	0.39^{a}	89.16^{b}
SLP02	0.99^{ab}	1.07^{cde}	3.49^{de}	5.20^{de}	0.53^{ab}	0.00^{a}	0.60^{a}	87.43^{ab}
SLP05	0.99^{ab}	1.00^{bcd}	3.02^{ab}	5.08^{cde}	0.66^{ab}	0.00^{a}	0.57^{a}	87.71^{ab}
SLP10	1.02^{ab}	0.78^{a}	2.92^{a}	5.01^{bcde}	0.59^{ab}	0.00^{a}	0.46^{a}	87.75^{ab}
SLP11	0.97^{ab}	1.03^{bcde}	3.17^{abc}	4.74^{bcd}	0.58^{ab}	0.00^{a}	0.49^{a}	88.68^{b}

The fatty acids of methyl esters in the same column followed by the combination of same or different letters did not show significant differences (≤0.01%)

Table 2.25 An analysis of the quality properties of the biodiesel samples.

Genotype	Content of H_2O (ppm)	Density (kg/m^3)	Viscosity (mm^2/s)					Acidity index	Iodine value	LHV (kJ/kg)	CN	CFPP (°C)				
			B100	B5	B10	B15	B20					B100	B5	B10	B15	B20
NL1M3	1824.4	968.0	77.2					1.2	85.6	37668.0	60.4	15				
NL2M1	1813.8	939.0	19.0					1.0	85.3	37695.0	60.5	15				
NL2M4	815.8	935.0	17.3	3.3	3.3	3.5	3.6	0.8	85.7	37697.0	60.4	15	−7	−6	−6	−7
NL2M6	6088.2	974.0	54.7					1.1	85.0	37626.6	60.5	15				
NL2M7	1532.6	970.0	53.8					1.1	85.3	37663.5	60.5	15				
Chi02	2612.5	948.0	19.2	3.3	3.7	4.1	4.6	1.0	85.4	37682.3	60.4	15	−8	−4	−5	−7
Edomex	1702.9	939.0	24.3	3.3	3.7	3.8	4.2	1.6	84.9	37659.7	60.6	15	−8	−5	−7	−7
SLP02	1488.8	934.0	17.4					0.8	85.8	37679.6	60.3	15				
SLP05	1356.0	930.0	15.8					1.1	85.9	37664.2	60.3	15				
SLP10	1414.5	988.0	158.3					1.3	85.9	37650.7	60.3	15				
SLP11	1331.4	936.0	18.3					1.2	85.4	37654.4	60.4	15				

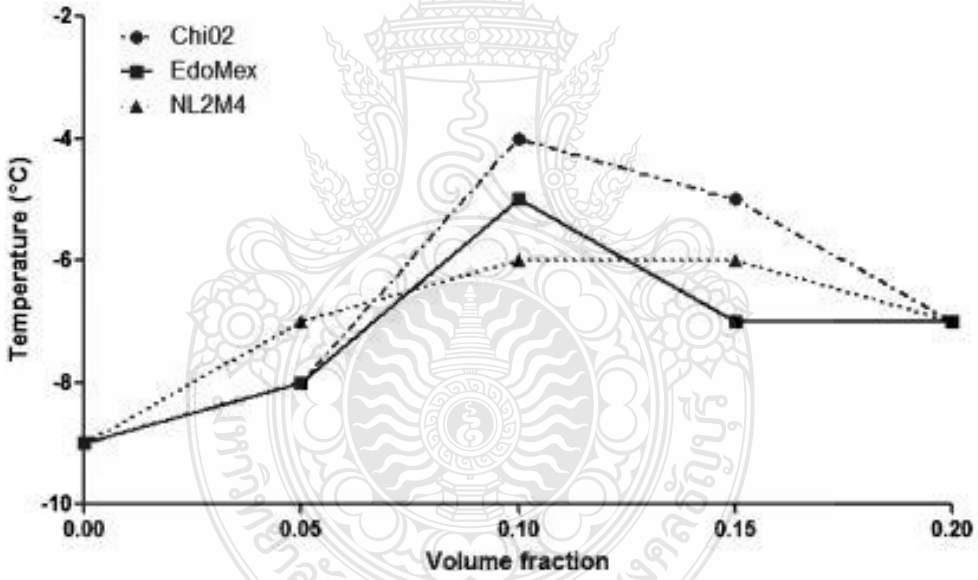


Figure 2.30 CFPP of B05, B10, B15 and B20 blends of biodiesel from three selected castor genotype samples.

Efthymia Alexopoulou et al. [38] as in Table 2.21, studied the fatty acid composition and oil content of castor seeds and found out that all the hybrid have a high oil content which ranges from 45.5% (C-1008, 2014) to 52.1% (Kaiima 75,2011). Ricinoleic acid was found to be the most dominant fatty acid in castor oil (12-hydroxy. 9 octadecenoic acid) with a percentage variation ranging from 84% (Kaiima 93, 2014) to 86.87% (Kaiima 93, 2011). The outcomes regarding the amount of oil in the seeds and the percentage of ricin oleic acid are following Wang et al's report, 2011 of (48% oil content and 85% ricin oleic acid of seed oil). Table 5 shows that there are other important fatty acids in castor oil, aside from ricin oleic acid which include; oleic acid, linoleic palmitic acid and stearic acid. The sum total of oleic and linoleic acid is 7.58% on average. The hybrid and overall trials range between 7.01% (C-854 in 2014) and 8.75% (C-1008 in 2014). This outcome shows how this is quite consistent with the report by Fernández, Martínez and Velasco (2012) and Wang et al (2011), which show that the combined effects of the two fatty acids in castor oil are around 10%.

Table 2.26. Fatty acid profile (Aliartos-GR) and oil content of seeds.

Hybrids/years		Oil content (%)	Oil profil					
			Ricin oleic acid (%)	Oleic acid (%)	Linoleic acid (%)	Palmitic acid (%)	Stearic acid (%)	Other fatty acids (%)
2011	Kaiima 71	48.90	84.80	3.16	4.01	1.89	2.03	4.11
	Kaiima 75	52.10	84.60	3.64	4.12	1.13	1.47	5.04
	Kaiima 93	46.60	86.87	3.08	4.12	1.51	1.46	2.96
	Mean	49.20	85.42	3.29	4.08	1.51	1.65	4.04
2012	Kaiima 93	48.80	85.36	3.12	4.09	2.08	2.09	3.26
	C-854	49.70	86.36	3.54	4.37	1.64	1.76	2.33
	C-855	48.90	86.31	3.14	4.18	1.33	1.53	3.51
	C-856	46.70	85.43	3.11	4.19	1.14	1.65	4.48
	C-864	45.80	86.28	3.2	4.17	1.77	2.01	2.57
	Mean	47.98	85.95	3.22	4.20	1.59	1.81	3.23
2014	Kaiima 93	49.50	84.00	3.74	4.73	1.57	1.77	4.19
	C-854	47.20	86.28	3.00	4.01	1.04	1.49	4.18
	C-855	46.60	85.63	3.63	4.19	1.81	1.57	3.17
	C-856	48.70	85.58	3.20	4.21	1.72	1.35	3.94
	C-1002	48.70	84.92	3.19	4.25	1.34	2.04	4.26
	C-1008	45.50	84.45	3.85	4.90	1.26	1.89	3.65
	Mean	47.70	85.14	3.44	4.38	1.46	1.69	3.90

Table 2.27 Calorific value and proximate ash and environmental analysis of harvested plants, Aliartos-GR in 2011 and 2012.

Years	Hybrids	Calorific value		Proximate analysis			Elemental analysis		
		GCV (MJ kg^{-1})	NCV (MJ kg^{-1})	Ash (%)	Fixed carbon (%)	Volatiles (%)	C (%)	H (%)	N (%)
2011 Leaves	Kaiima 71	17.74	16.56	13.84	10.66	75.51	41.44	5.57	2.63
	Kaiima 75	17.43	16.27	14.07	11.74	74.20	41.24	5.51	3.52
	Kaiima 93	16.90	15.80	13.72	11.42	74.87	42.15	5.23	2.81
	Mean	17.36	16.21	13.88	11.278	74.86	41.61	5.44	2.99
Stems	Kaiima 71	17.04	15.88	9.05	13.54	77.42	41.64	5.51	0.45
	Kaiima 75	17.75	16.59	7.12	15.22	77.66	41.24	5.53	0.41
	Kaiima 93	17.30	16.16	9.09	13.17	77.75	42.09	5.43	0.80
	Mean	17.36	16.21	8.42	13.98	77.61	41.66	5.49	0.55
2012 Whole plant	Kaiima 93	16.96	15.75	9.21	13.91	76.89	42.21	5.71	0.94
	C-854	16.98	15.76	9.93	13.17	76.91	41.84	5.73	0.79
	C-855	16.97	15.77	9.43	13.98	76.60	42.74	5.70	0.73
	C-856	16.92	15.78	11.47	12.68	75.85	41.50	5.41	0.95
	C-864	16.65	15.48	11.75	12.61	75.64	41.40	5.55	0.81
	Mean	16.90	15.71	10.36	13.27	76.38	41.94	5.62	0.8

S. N. A. M. Hassan et al. [39] carried out a comparative study on rubber seed shell and kernel (Hevea Brasiliense) as raw material for the production of bio-oil. This study dealt mostly on the characteristics of two plant material samples, which are; rubber peelings (RSS) and rubber kernel (RSK) as major raw material for biofuel production. The biomass samples used were collected from Kedah- Malaysia. A thorough check was conducted to ascertain their physical and chemical characteristics as well as composition, such as, extracts, amount of holocellulose and hemicellulose. The calorific value, oil yield, and pyrolysis in both samples was also considered. The result shows that the carbon and volatile content of RSK is high up to (64.5 wt%) and (92.4 wt%) respectively. The total biomass had a calorific value ranging from 23 MJ / kg to 27 MJ / kg. From the study, it can be deduced that rubber seeds could be an alternative source of biofuel production.

Table 2.28 Analysis of the ultimate biomass samples.

Biomass	C	H	N	S	O*
RSS	48.8	5.9	1.5	0.1	43.7
RSK	64.5	8.2	3.6	0.3	23.4

*Calculated by difference

Table 2.29 Analysis of the proximate biomass samples

Biomass	Moisture	Ash	Volatile Matter	Fixed Carbon
RSS	14.3	0.1	71.7	13.9
RSK	4.3	0.2	89.4	6.1

Table 2.30 The content of the biomass samples

Biomass	Extractives	Holocellulose	Hemicellulose	*Cellulose
RSS	7.8	92.2	66.4	25.8
RSK	3.6	96.4	26.9	69.5

*Calculated by difference

Table 2.31 Calorific value and yield of the biomass samples

Biomass	Calorific Value (MJ/kg)	Oil Yield (wt %)
RSS	23.9	8.7
RSK	27.5	33.1

Kaushik K. Dhar Dubey et al. [40] collected seed samples from three different geographic locations in India. The total amount of fat and lignocellulose content of seeds ranged from 54 to 69% and 69–87% respectively. A noteworthy property of the

H. Bengalese seed oil (HbSO), is that it is inedible, but it possesses credible potentials to be used as a biodiesel. Through a process of transesterification, seed oil is converted into biodiesel ~93%. From the analysis of its fuel properties such as acid value, heat value, viscosity, density, ash content, flash, pour and cloud point of biodiesel according to ASTM-D6751 Standards as well as comparisons with other biodiesel properties, the report shows reasonable quality. Therefore, this oil that is not edible possesses the potential to be used as an energy source for the production of biodiesel.

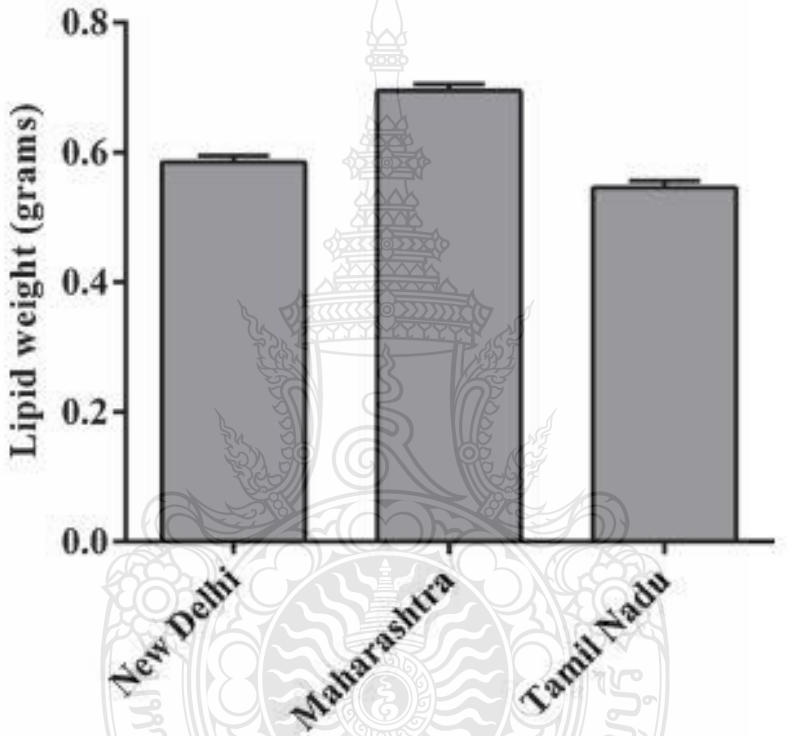


Figure 2.31 Total lipid weight per gram of mature H. Bengalese seeds collected from different locations in India.

Table 2.32 Total lipid and FA content of mature H. Bengalese seeds collected from different locations in India.

Fatty acid	New Delhi	Maharashtra	Tamil Nadu
Palmitic acid	2.42 ± 0.31	4.58 ± 0.26	2.46 ± 0.33
Stearic acid	1.34 ± 0.12	3.39 ± 0.06	2.28 ± 0.14
Oleic acid	2.14 ± 0.09	12.11 ± 4.38	2.29 ± 0.38
Linoleic acid	6.51 ± 0.35	10.66 ± 2.98	6.22 ± 1.22
Ricinoleic acid	87.5 ± 0.82	69.06 ± 7.18	86.71 ± 1.33
Total Lipid weight (g/g of seed)	0.585 ± 0.01	0.695 ± 0.01	0.546 ± 0.01

The values represent the mean ± SDs of three samples.

Table 2.33 Physical and chemical properties of H. Bengalese seed oil.

Parameters	HbSO1	HbSO2
Percentage oil content	58.5 ± 2.4	46 ± 2.49
pH	6.5-7	6.5-7
Colour	Dark Yellow	Pale Yellow
Physical state at room temperature	Liquid	Liquid
Density (g/mL)	0.964 ± 0.02	0.870 ± 0.02
Free fatty acid (mg KOH/g)	3.76 ± 0.4	1.78 ± 0.13
Acid value (mg KOH/g)	7.48 ± 0.8	3.54 ± 0.26
Iodine value (g/100 g)	108.7 ± 3.8	108.7 ± 2.60
Saponification value (mg KOH/g)	238 ± 4.49	204 ± 4.49

*The values represent the mean ± SDs of three samples.

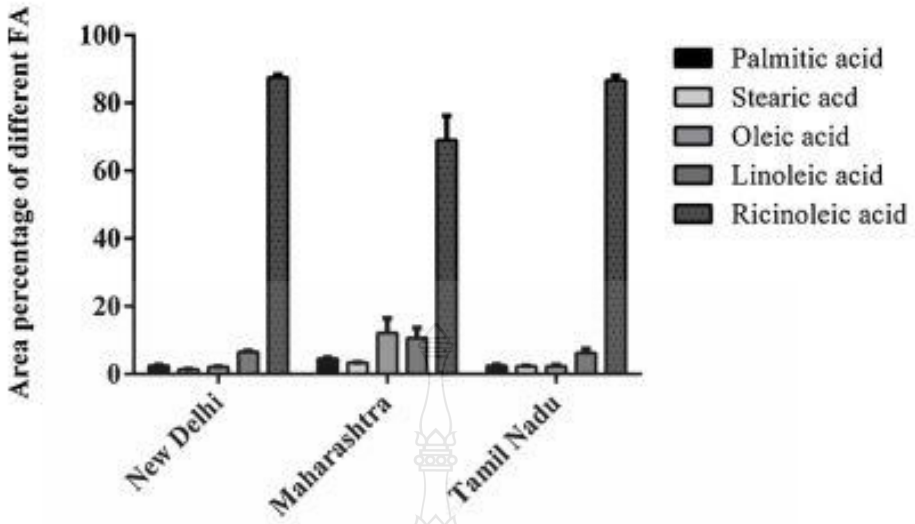


Figure 2.32 Shows percentage area of 5 major FA components of mature H. Bengalese seeds collected from different locations in India.

Table 2.34 Physical and chemical properties of H. Bengalese biodiesel.

Parameters	Hiptage biodiesel	Castor biodiesel (Bateni et al., 2014)	Jatropa biodiesel (Amalia et al., 2013)	ASTM-D6751 (Amalia et al., 2013; Bateni et al., 2014)
Flash point (^{0}C)	49.33 ± 4.02	147 ± 3	107	130 (minimum)
Cloud point (^{0}C)	12.33 ± 2.05	−14 ± 1	11	18 (minimum)
Pour point (^{0}C)	−14.66 ± 2.40	–	0	0 (maximum)
Kinematic viscosity at 40^{0}C (mm^2/s)	4.47 ± 0.24	16.3 ± 0.8	3.5	1.96-6.0
Density at 15^{0}C (Kg/m^3)	836.66 ± 3.68	–	885	850-890
Acid number (mg KOH/g)	0.7 ± 0.14	0.48 ± 0.04	0.35	0.8 (maximum)
Iodine value (g/100 g)	70.69 ± 1.58	–	107	115 (maximum)
Saponification value (mg KOH/g)	141.8 ± 2.64	–	–	–
Refractive index	1.46	–	–	–
Water content (%)	0.91 ± 0.43	0.03 ± 0.01	Trace	0.05
Ash content (%)	~0	–	–	0.01(maximum)
Calorific value (MJ/kg)	33.12 ± 0.23	35.2 ± 1.2	40	35 (minimum)

*The values represent the mean ± SDs of three replicates.

Debashis Sut et al. [41] Study of seeds of biofuel production through chemical and thermal chemical transformation pathways. The biodiesel properties were compared with those derived from similar oil seeds and it showed that the by-products from the

chemical processes could be used as raw materials for pyrolysis at different temperatures in a fixed bed reactor. In a temperature of 500ºC, the highest bio-yields of between 29.11% and 26.18% were found. The highest bio-oil yields obtained were CHN, NMR and FTIR spectroscopy analyzers. The biological properties are characterized by SEM - EDX. XRD and FTIR together with component analyzed to investigate its use presently. The result shows a new way to make full use of fat-rich biological resources to different things.

Table 2.35 Profile of fatty acid C. thevetia biodiesel.

Fatty acid	Formula	Structure	Retention time (in min)	wt.%
Palmitic acid	$C_{16}H_{32}O_2$	$C_{16:0}$	33.75	20.67
Stearic acid	$C_{18}H_{36}O_2$	$C_{18:0}$	36.70	12.42
Oleic acid	$C_{18}O_{34}O_2$	$C_{18:1}$	36.49	43.92
Linoleic acid	$C_{18}H_{32}O_2$	$C_{18:2}$	36.39	20.76
Arachidic acid	$C_{20}H_{40}O_2$	$C_{20:0}$	39.83	2.21

Table 2.36 Properties of oil and FAME Cascabela thevetia compared with some non-edible oil seeds.

Properties	*Sapindus mukorossi* (Chakraborty and Baruah, 2013)		*Terminalia bellerica* (Chakraborty et al., 2009)		*Pongamia glabra* (Chakraborty et al., 2009)		*Jatropha curcas* (Raheman, 2012)		*Cascabela thevetia* [present study]		ASTM D6751-07	EN14214-07
	Oil	FAME	Oil	FAME	Oil	FAME	Oil	FAME	Oil	FAME		
Density (kg/m^3)	923	876	910	882	931	903	940	880	898	882	NA	860–900
Kinematic viscosity (mm^2/s)	32.10	4.63	25.60	5.17	26.06	6.13	24.50	4.80	28.71	5.11	1.9–6.0	3.5–5.0
Calorific value (MJ/kg)	38	40.02	37.50	39.22	40.51	43.42	38.65	39.23	39.33	39.76	NA	NA
Cetane number	NA	56	NA	53	NA	55	NA	NA	NA	57	47 (min)	51 (min)
Acid value (mg KOH/g)	15.6	0.14	12.50	0.23	1.21	0	28.00	0.40	13.24	0.41	0.80 (max)	0.50 (max)
Induction period (hr.)	NA	1.20	NA	NA	NA	NA	NA	NA	10.38	5.84	3	6
Cloud point (°C)	NA	−1	NA	NA	NA	NA	NA	NA	8	6	NA	−3 to 12
Pour point (°C)	6	−4	3	6	0	−8	4	2	−5	−8	NA	−15 to 10
Flash point (°C)	159	140	102	90	NA	95	NA	135	ND	130	130 (min)	120 (min)
Ash content (wt.%)	0.02	0.003	0.0012	0.0005	0.001	0.001	0.8	0.012	0.05	0.003	0.02 (max)	0.02 (max)

Table 2.37 Properties of biomass and CTDC and CTSC biochar.

Properties	CTDC				CTSC			
	Biomass	Biochar			Biomass	Biochar		
		400 °C	500 °C	600 °C		400 °C	500 °C	600 °C
pH	-	7.40	10.80	11.20	-	8.68	9.70	10.21
EC (mʊ)	-	0.96	1.13	1.74	-	0.84	0.98	1.21
Moisture content	5.10	4.80	4.20	3.80	3.60	2.70	1.80	1.40
Ash content	4.20	7.90	9.35	10.45	4.52	9.92	10.85	12.28
Volatile content	73.81	19.35	12.70	10.55	75.71	20.75	17.80	11.68
Fixed carbon	16.82	65.87	70.20	72.70	16.15	68.25	71.20	74.82
C	46.08	58.38	64.60	67.60	47.50	65.40	74.80	77
H	7.20	2.83	2.42	2.17	6.44	2.85	2.60	2.18
N	6.43	6.11	5.77	4.95	1.75	1.15	1.67	1.50
O	40.29	32.68	27.21	25.28	44.31	30.60	20.93	19.32
H/C molar ratio	1.87	0.58	0.45	0.39	1.63	0.52	0.42	0.34
O/C molar ratio	0.59	0.42	0.32	0.28	0.93	0.35	0.21	0.19
Calorific value (MJ/kg)	19.25	23.80	25.87	26.65	20.65	24.52	26.76	27.65
R_{50} index	-	0.58	0.61	0.68	-	0.52	0.55	0.61
CSP %	-	23.62	26.24	32.21	-	21.34	25.10	30.10

Table 2.38 Physio - chemical properties of bio-oil compared with petro-fuels.

Properties	Methods	CTDC bio-oil	CTSC bio-oil	PGDC bio-oil (Chutia et al. 2014)	PGSC bio-oil (Bordoloi et al. 2015)	Gasoline	Diesel
Appearance		Dark brownish color	Dark brownish color	Dark brownish color	Dark brownish color	-	-
Density at 15 °C (kg/m^3)	ASTM D 1298-99	962	978	1051	958	0.72-0.78	0.82-0.85
Specific gravity at 15 °C/15 °C	ASTM D 1298-99	963	979	-	958.3	-	-
pH		3.6	3.8	-	3.9	-	-
Pour point (°C)	ASTM D 5853-01 (2007)	+5	+7	-	+4	−40	−40 to −1
Cloud point (°C)	ASTM D 1310-99	18	23	-	15	-	-
Flash point (°C)	ASTM D 6450-05 (2010)	44	48	-	42	−43	53–80
Fire point (°C)	ASTM D 1310-01 (2007)	56	61	-	58	-	-
C		55.76	59.78	55.4	56.12	-	-
H		7.39	7.68	7.8	6.97	-	-
N		4.77	9.61	5.7	1.83	-	-
O		32.08	22.93	31.10	31.87	-	-
H/C		1.59	1.54	1.69	1.49	-	-
O/C		0.43	0.29	0.56	0.43	-	-
Empirical formula		$C_{13.68}H_{21.73}NO_{5.91}$	$C_{7.22}H_{11.13}NO_{2.07}$	$C_{11.34}H_{19.16}NO_{4.77}$	$C_{35.7}H_{53.3}NO_{15.2}$	-	-
Calorific value (MJ/kg)		27.86	28.35	28.19	29.45	42–46	42–45

Table 2.39 Percentage of hydrogen from 1H NMR analysis of CTDC and CTSC bio-oil.

Proton assignment	Chemical Shift (ppm)	CTDC bio-oil	CTSC bio-oil
Aromatics	6.5–9.0	17.07	18.82
Phenolic OH or olefinic proton	5.0–6.5	15.87	12.89
Ring-join methylene (Ar–CH_2–Ar)	3.3–4.5	12.79	15.55
CH_3, CH_2, CHα to an aromatic ring	2.0–3.3	29.92	26.27
CH_2, CHα to an aromatic ring (naphthenic)	1.6–2.0	5.83	4.67
β-CH_3, CH_2, and CHγ or further from an aromatic ring	1.0–1.6	14.16	19.09
CH_3γ or further from an aromatic ring	0.5–1.0	4.95	3.31

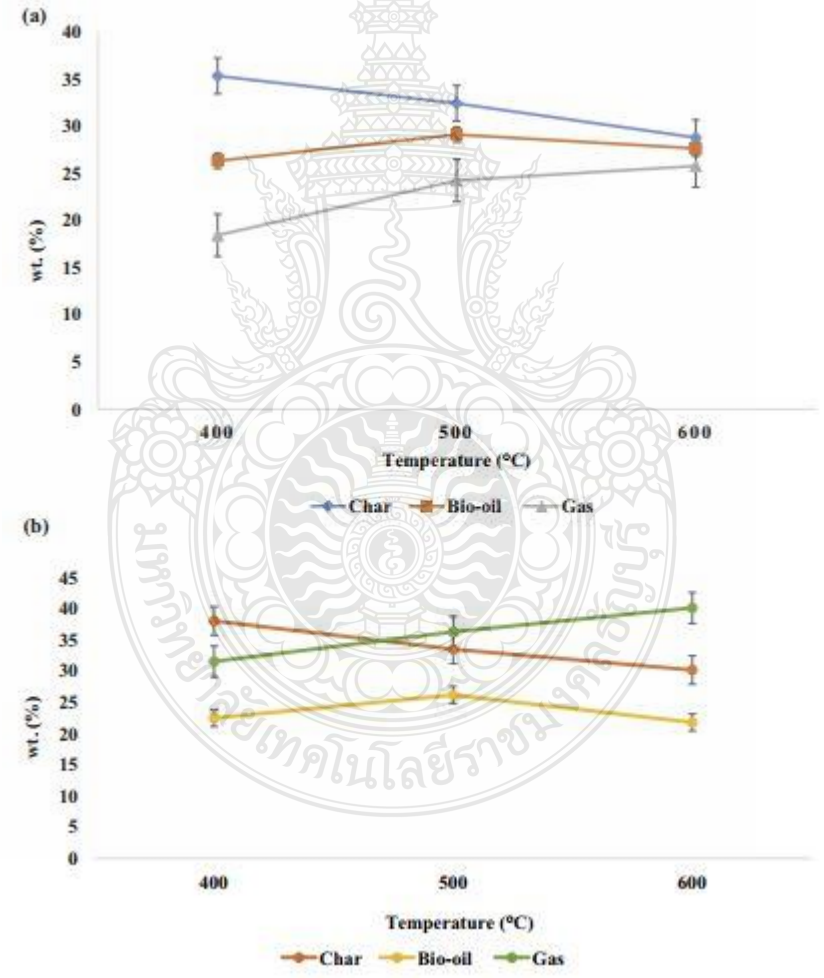


Figure 2.33 (a) Yields of CTDC biomass. (b) Yields of CTSC biomass.

Ahmed, N.B. et al (2016) [42] investigated biodiesel production from roselle oil seeds and the determination of optimum reaction conditions for the transesterification process. The research aimed at extracting oil from roselle seeds and then using it as a sample to investigate biodiesel fuel production as well as the optimal condition for the alkali-catalyzed transesterification process. For the experimental research, the transesterification method represented as: RCOOR + R'OH ⇆ R'COOR' + ROH and methanolysis was adopted. The researchers made use of ground roselle seeds, hexane, methyl alcohol with 20ºC (0.79) density and minimum assay (GC) 99.0%, methyl orange, sodium hydroxide as the alkaline base, and potassium hydroxide with minimum assay (GC) 85.0% as the catalyst. For the oil extraction process, 1000g of roselle seed powder was deposited in a cellulose paper cone Soxhlet and the oil was extracted with n-hexane in eight hours. The solvent and n-hexane were evaporated leaving behind 191.1g of roselle oil which when analyzed by gas chromatography had 0.907g/mL density, 257.18g/mol average molecular weight, and 19.11% of oil percentage. The roselle oil was then subjected to transesterification with methanol by using potassium hydroxide (KOH) as a catalyst. The analysis of fatty acid composition in the roselle oil by gas chromatography was as below.

Table 2.40 Roselle seed oil composition, oil percentage and molecular weight in (red calyces) by GC

Peak no.	Name	Percentage (%)	Molecular weight g/mol
1	Decanoic acid	3.6926	178.26
2	Pentadecanoic acid	12.0982	242.3975
3	Pentadecenoic acid	35.2829	242.3975
4	Palmatic acid	33.2440	256.4241
5	linolenic acid	0.1965	278.44
6	linolenic acid	0.5261	278.44
7	Linolelaidic acid	1.9506	280.45
8	Oleic acid	1.4421	282.4614
9	Arachidic acid	2.2646	304.4669
10	Eicosatrienoic acid	3.9613	306.48276
11	Nervonic acid	5.4310	366.62
Total	11	100.0	Sum Mwt =257.18

Much higher values of fuel properties especially kinematic viscosity and density were found in the roselle oil which made its direct usage as fuel in diesel engines impossible. These values were reduced to permissible levels after transesterification. The cetane number, sulfur content, and total acid number were found at good biodiesel standing as shown in the comparison with ASTM specifications below.

Table 2.41 Properties of Roselle biodiesel in comparison with ASTM specifications

Property	Test method	Unit	Roselle biodiesel	Biodiesel specification(ASTM)
Density at 15 ^{0}C	ASTM D4052	g/mL	0.8829	--
Pour point	ASTM D97	^{0}C	+3.0	-15-10
Flash point	ASTM D93	^{0}C	172.0	Min. 130
Kinematics viscosity at 40 ^{0}C	ASTM D445	cSt	5.320	1.9-6
Sulfur content	ASTM D4294	mass%	0.0255	Max. 0.02
Total acid number (TAN)	ASTM D664	mg KOH/g	0.04	Max. 0.5
Cetane number	Chart	--	53	48-65

The optimum of reaction variables was determined by considering any one of the reaction time, reaction temperature, catalyst concentration, and oil to methanol molar ratio when others were constant. After the attainment of an optimum, the value was maintained constant, and then the optimum of the next variable was determined. The biodiesel yield percentage were as follows:

From the observed effect of variation of catalyst amount on yield percentage, the best result was obtained at 0.5 wt % catalyst amount which yielded 90.9%. For the effect of variation time on yield percentage, 40 minutes had the best result with a 90.9% yield. That of variation of reaction temperature on yield percentage had the best outcome at 65°C with a 99.5% yield. The methanolysis of roselle oil stoichiometrically requires three moles of methanol for each mole of oil. The best result obtained a 93% yield at 1:3. In conclusion, the study demonstrated that roselle oil can successfully produce biodiesel through an alkali-catalyzed transesterification reaction with methanol in the presence of potassium hydroxide (KOH) as a catalyst. The optimal producing

conditions were catalyst concentration of 0.5 wt %, reaction temperature of 65°C, 1:3 molar ratio of oil to methanol, and a reaction time of 40 minutes.

Sahu, A et al (2017) [43] undertook a study on performance and experimental analysis of roselle oil as biodiesel blend on four strokes diesel engine. The study aimed to convert roselle oil to biodiesel by the process of transesterification, compare their physicochemical properties with those of petroleum-based diesel, and test the performance of the biodiesel from these oils at various blend ratios with diesel using the Compression Ignition (CI) Engine. The experimental investigation employed the use of roselle seeds, potassium hydroxide (KOH) as alkaline catalysts with methanol (CH3OH) for the production of biodiesel. The transesterification process involved mixing of alcohol and catalyst, reaction, separation of glycerin and biodiesel, methyl ester washing, and heating of biodiesel. 350ml biodiesel and 300ml glycerol were obtained from 500ml roselle oil with 150ml methanol and 10g of potassium hydroxide. The roselle biodiesel was blended with diesel, methanol, and ethanol at various proportions and tested on a CI engine. Blend A had 5% Methanol, 5% roselle biodiesel, and 90% Diesel; Blend B had 5% Methanol, 10% roselle biodiesel, and 85% Diesel; Blend C had 5% Methanol, 20% roselle biodiesel, and 75% Diesel; Blend D had 5% Methanol, 30% Roselle biodiesel, 65% Diesel; Blend E had 5% Ethanol, 5% Roselle biodiesel, and 90% Diesel; Blend F had 5% Ethanol, 10% Roselle biodiesel, and 85% Diesel; Blend G had 5% Ethanol, 20% Roselle biodiesel, and 75% Diesel; and Blend H had 5% Ethanol, 30% Roselle biodiesel, and 65% Diesel. The properties of the blends were as follows:

Table 2.42 Properties of eight different blends

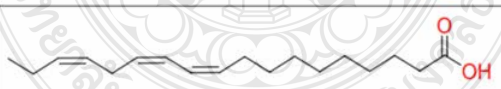
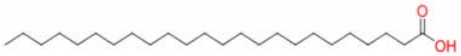
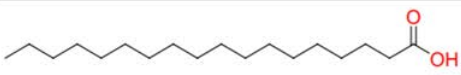
Type of Blend	Amount of Methanol/Ethanol over 1000 ml	Amount of Biodiesel over 1000 ml	Amount of Diesel over 1000 ml	Density (kg/m^3)	Calorific value (KJ/kg)
Diesel	-------	-------	1000	840	43910.00
Blend A	50	50	900	850.1	41968.67
Blend B	50	100	850	849.7	41238.83
Blend C	50	200	750	848.3	39779.25
Blend D	50	300	650	842	38319.70
Blend E	50	50	900	850.2	42324.46
Blend F	50	100	850	849.5	41594.67
Blend G	50	200	750	848.1	40135.10
Blend H	50	300	750	841.8	38675.53

The research findings revealed that the brake power of blends A, B, C, and D had approximately the same value and was almost like that of diesel. All the biodiesel blends and diesel fuel had the same trend of brake-specific fuel consumption but differ in middle and higher load conditions. The brake-specific energy consumption for blends B, C, and D was less than diesel at low load conditions; Blend D was less when compared with others at middle loads; Blend A showed least at higher loads. The brake thermal efficiency of Blend A was less than that of blends B, C, and D; lesser than diesel at middle load but highest at higher load conditions. With regards to brake power, all biodiesel blends and diesel had the same trend; but Blend H had a higher value than diesel and blends E, F, and G at higher load conditions. For brake-specific fuel consumption, pure diesel had the least consumption as compared to blends E, F, G, and H; with Blend H closest to diesel at higher load conditions. On brake specific energy consumption, all blends of biodiesel had the same trend with diesel at low load conditions; but Blend H was least at middle and higher load conditions when compared with diesel and blends E, F, and G. With regards to brake thermal efficiency, Blend H had the highest efficiency at load increase as compared to diesel and blends E, F, and G. In conclusion, Blend A had better performance than blends B, C, and D when compared with diesel fuel; with the least brake specific fuel and energy consumption and highest brake power and brake thermal efficiency. Blend H exhibited better performance amongst blends E, F, and G in comparison with diesel fuel; with the least brake specific

energy consumption, and highest brake power and brake thermal efficiency except for pure diesel which had better brake specific fuel consumption than other blends. Also, roselle biodiesel was found to be superior to other biodiesels. Its mixture with diesel and 5% of either methanol or ethanol increases diesel engine performance, especially at high load conditions.

Verma et al. (2021) [44] undertook an experimental and empirical investigation of a CI engine fueled with blends of diesel and roselle biodiesel. The study aimed at assessing the technical viability of roselle biodiesel and its binary mixes with diesel as a working fuel in a single-cylinder 4-stroke CI engine, examining the engine's overall features from the assessment result, and creating an artificial neural network-based empirical model. Through the transesterification process, 830ml roselle biodiesel was obtained from 1000ml roselle oil with 280ml of methanol at a 1:3 molar ratio and 1.65 ml sodium hydroxide (NaOH) between 60–75 °C at 60-70 minutes and 320–600 rpm. The fatty acid content of the roselle oil was analyzed with gas chromatography-mass spectrometry and its composition was as below.

Table 2.43 Fatty acid composition of Roselle biodiesel

Fatty Acid	Structure	Molecular structure	Formula	Mol. wt	% (w/w)
Linoleic acid	18:2		$C_{18}H_{32}O_2$	280.2512	38.18
Palmitic acid	16:0		$C_{16}H_{32}O_2$	257.2503	18.49
Linolenic acid	18:3		$C_{18}H_{30}O_2$	279.2235	2.08
Oleic acid	18:1		$C_{18}H_{34}O_2$	281.2667	33.32
Lignoceric acid	24:0		$C_{24}H_{48}O_2$	367.3543	1.11
Stearic acid	18:0		$C_{18}H_{36}O_2$	285.2624	4.08

The roselle biodiesel and diesel were blended at various proportions according to ASTM standards. Sample blends consisted of LA20 (Roselle 20%+Diesel 80%),

LA40 (Roselle 40%+Diesel 60%), LA60 (Roselle 60%+Diesel 40%), LA80 (Roselle 80%+Diesel 20%) and LA100 (Roselle biodiesel 100%), and their properties were as follows:

Table 2.44 Important physical-chemical properties of diesel, Roselle, and its blends.

Fuel	Testing method	B0	LA20	LA40	LA60	LA80	LA100
Density (@20°C) (kg/m^3)	ASTM D4052	830	838.2	849.6	859.2	868.6	877
Kinematic viscosity (@40°C) (mm^2/s)	ASTM D445	2.9	3.24	3.5	4.25	4.85	5.64
Heating value (MJ/ kg)	ASTM D4809	42.5	41.6	40.7	40.16	39.44	38.74
Cetane number	ASTM D613	48	48.8	49.67	50.45	51.25	52.3
Flash point (°C)	ASTM D93	50	75.2	94.4	116.7	135.2	159.2
C%	ASTM D5291	86.14	84	83.09	81.52	80.10	78.71
H%	ASTM D5291	13.86	13.49	13.12	12.75	12.29	12.12
O%	ASTM D5291	0.004	2.25	4.02	5.71	7.54	9.23

The roselle biodiesel and its blends were subjected to testing with a fixed compression ratio of 17.5:1, fuel injection timings of 19°, 21°, 23°, 25°, and 27° before top dead center, engine loadings of 25%, 50%, 75% and 100% and a constant engine speed of 1500rpm. Exhaust emissions were measured with a testo-350 gas analyzer. An artificial neural network was used to construct an empirical model for the prediction or optimization of the IC engine characteristics. From the experiment, it was found out that brake thermal efficiency dropped as fuel injection timing increased from 19° to 27° bTDC. When compared with other blends, LA20 had higher brake thermal efficiency with a decrease in fuel injection timing and lowers with an increase in the latter under higher load conditions. The brake thermal efficiencies of LA20 and LA40 were approximately identical to diesel fuel, but those of LA60, LA80, and LA100 were significantly lower. However, diesel fuel had higher brake thermal efficiency than roselle biodiesel and its blends. The brake specific fuel consumption increased as the fuel injection timing increased from 19° to 27° b TDC. Diesel fuel was found at a lower brake specific fuel consumption as compared to roselle and its blends. Retardation in fuel injection timing lowered exhaust gas temperature by 3-6°C, while its acceleration enhanced the latter for all tested fuels. A higher cylinder pressure for all engine operating conditions at different fuel injection timing was recorded for diesel fuel as compared to other biodiesel and its blends. It was also deduced that fuel injection timing

retardation reduced the ignition delay period, thereby resulting in a lower peak heat release rate and premixed combustion. The ignition delay period for diesel fuel was longer than for roselle biodiesel and it mixes at all operational fuel injection timings. Diesel fuel again exhibited a larger pressure rise than the other tested fuels in all engine running situations. With regards to the emission of smoke and nitrogen oxide, diesel fuel had more production, followed by roselle biodiesel and its blends; but lower in CO2 emission. In conclusion, the technological feasibility of roselle biodiesel makes it a good alternative to diesel fuel while its growability and commercial viability boost its significance economic wise.

Bothon et al (2020) [45] investigated the physicochemical variability and biodiesel potential of seed oils of two hibiscus sabdariffa L. phenotypes. It was a comparative study of the physicochemical properties of two varieties of hibiscus sabdariffa L. – red phenotype, sabdariffa (HSS) and green phenotype, altissima (HSA) aimed at highlighting their potential use as fuel. The quality parameters and fuel properties of HSS and HSA were as follows:

Table 2.45 Quality Indices of HSA and HSS Seed Oils

physicochemical parameters	HSA	HSS	diesel[20,17,21]	ASTM D6751[17]	EN 14214[17]
extraction yield (%)	16.01 ± 0.06a	15.34 ± 0.25b			
density 20 ° C (g/cm^3)	0.876 ± 0.001a	0.875 ± 0.001a	0.845	0.88	0.86–0.90
acid number (mg KOH/g oil)	23.10 ± 0.22a	18.20 ± 0.40b	0.17	max 0.5	max 0.5
cetane number	55.376 ± 0.875a	55.515 ± 0.246a	45–55	min 47	min 51
iodine index (g I_2/100 g oil)	90.449 ± 2.960a	90.988 ± 1.665a	6		120
peroxide value (mequiv O_2/kg oil)	0.280 ± 0.001a	0.140 ± 0.001b			
saponification index (mg KOH/g oil)	145.78 ± 2.01a	132.33 ± 2.40b			
refraction index	1.468 ± 0.001a	1.468 ± 0.001a			
kinematic viscosity 20 °C (mm^2/s)	3.820 ± 0,027a	3.833 ± 0,006a	2.91–4	1.9–6.0	3.5–5.0 (40 °C)
higher heating value (MJ/kg)	39.450 ± 0.007a	39.459 ± 0,010a	42–45.9	35	35
oxidation stability (h)	6.286 ± 0.352a	6.418 ± 0.124a			min 6
cold filter plugging point (°C)	−16.265 ± 0.011a	−16.267 ± 0.003a			

[a]Means not sharing a common letter in the same row denote a significant difference at $P < 0.05$

It was discovered that HSA had more significance than HSS in terms of oil yields. However, there was no significant difference between their density (~0.87), kinematic viscosity (~3.820 mm2/s), higher heating value (~39.45 MJ/kg), and cetane number (~55.37). Their kinematic viscosity was low and within the U.S and European standards range. Though they released less energy than diesel, their released energy was

nevertheless beyond the U.S. and European recommended minimum value of 35 MJ/Kg. With regards to their cetane numbers, they were almost at the maximum value obtained with diesel (45−55) and beyond the standard value of min 47 and min 51. Their iodine number complies with the EN14214 biodiesel standard which has the maximum value of 120 g I2/g oil, and indicates the predominance of monounsaturated fatty acids. A significant difference was recorded in their acid numbers (23.10 ± 0.22 vs 18.20 ± 0.40mg KOH/g oil for HSS) where HSA had a higher number. The difference was attributed to the degradation of chlorophyll in the seeds as degradation of dyes releases acidic substances. Their seed oils were also different in color as that of HSS was yellow and HSA was dark green. This variation was linked with a variety in pigment contents such as anthocyanins and chlorophyll. Albeit their peroxide values met the normative requirement of (<10 mequiv O2/kg oil, the two oils were significantly different (HSA: 0.280 ± 0.001 vs HSS: 0.140 ± 0.001). It means that both oils could be stored without fear of major alteration. Again, both oils were different in saponification numbers (HSA: 145.78 ± 2.01 mg KOH/g oil vs HSS: 132.33 ± 2.40 mg KOH/g oil). The high value presupposes the presence of a large amount of fatty acid used in soap fabrication in the transesterification process. The oxidation stability for both oils was reached at the minimum value of 6 h for the induction period at 110 °C and measured with the standard recommended Rancimat method. Both HSA and HSS had the same cold filter plugging point of −16.26 °C. Furthermore, the chromatograms of both oils were almost identical and stackable as depicted in the figures below.

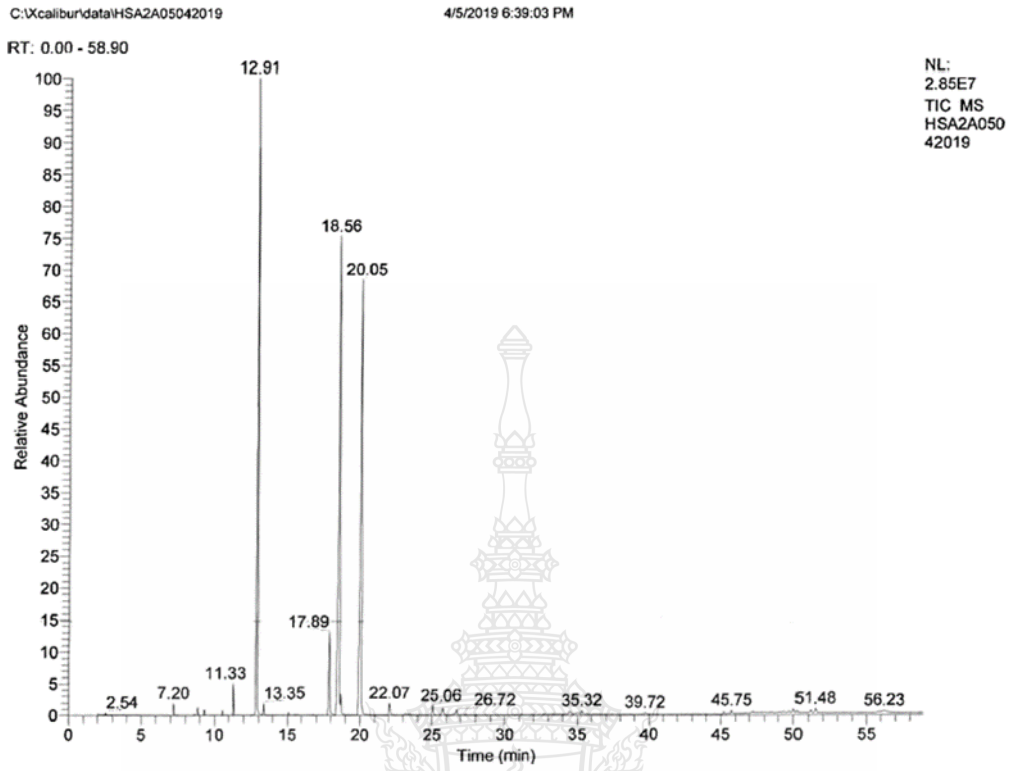


Figure 2.34 HSA and HSS chromatogram.

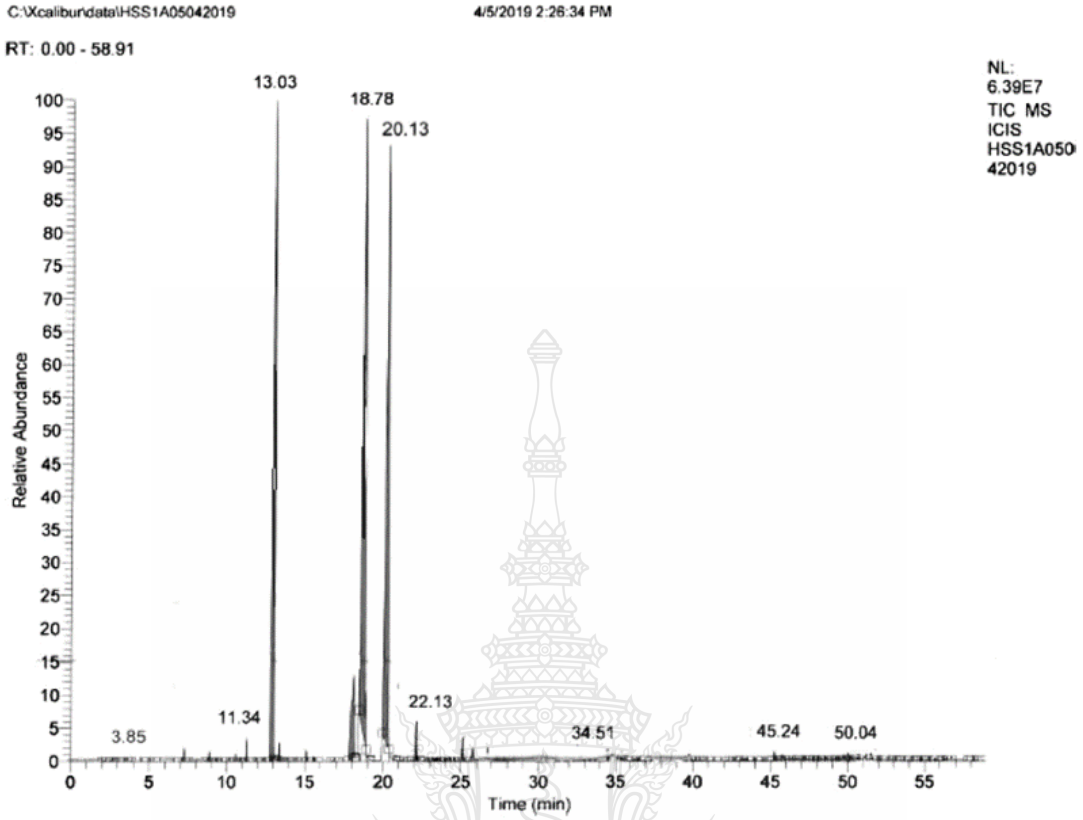


Figure 2.35 Gas chromatogram of fatty acids in HSS seeds.

Three intense peaks were recorded at the retention times of 12.91 (13.03), 18.56 (18.78), and 20.05 (20.10), and these corresponded to palmitic, oleic, and linoleic acids as seen in the following figure.

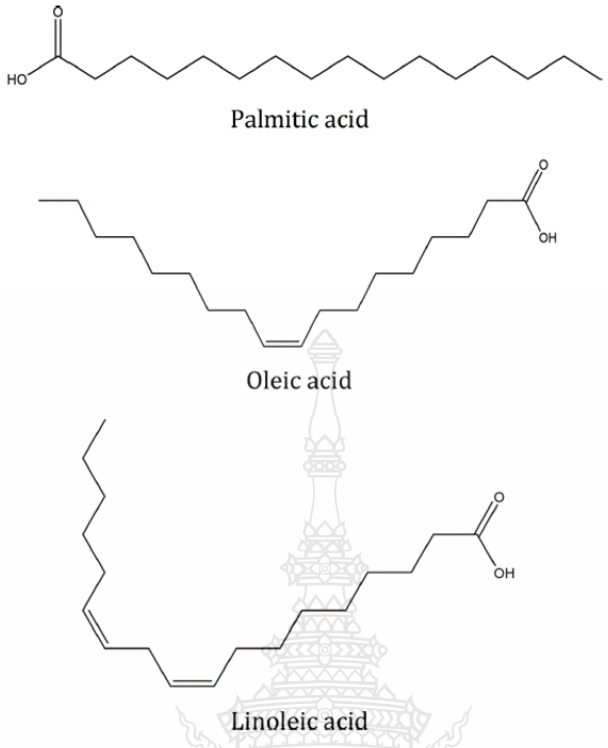


Figure 2.36 Major fatty acids of the two oils

The fatty acid composition of HSS and HSA were fairly balanced: saturated fatty acids (HSA: 33.65% and HSS: 32.78%, m/m), monounsaturated fatty acids (HSA: 33.86 and HSS: 36.29%, m/m), and polyunsaturated fatty acids (HSA: 32.1 and HSS: 30.83%, m/m) but different in some aspects. There was a lack of myristic and nervonic acids in HSA seed oil, and eicosenoic and lignoceric acids in HSS seed oil. The table below outlines the complete compositions of both oils.

Table 2.46 Fatty Acids Composition (%) of HAS and HSS Seed Oils

free fatty acid		HSA	HSS	works on HSS[1,24,25]
tridecanoic	C13:0	0.30	0.23	
myristic	C14:0		0.15	0.23–0.31
pentadecenoic	C15:1	0.86	0.36	
palmitic	C16:0	27.09	25.48	18.15–21.65
palmitoleic	C16:1	0.38	0.26	0.44
stearic	C18:0	5.01	5.54	4.09–5.47
oleic	C18:1	31.81	35.21	30.90–38.46
linoleic	C18:2	31.43	29.7	38.17–40.12
linolenic	C18:3	0.67	1.13	0.57–2.09
arachidic	C20:0	0.56	0.69	0.72
eicosenoic	C20:1	0.54		0.08
behenic	C22:0	0.33	0.44	0.37
erucic	C22:1	0.27	0.29	
tricosylic	C23:0	0.18	0.25	
lignoceric	C24:0	0.18		
nervonic	C24:1		0.17	
total SFA		33.65	32.78	
total MUFA		33.86	36.29	
total PUFA		32.1	30.83	

The prepotent saturated fatty acids were palmitic and stearic, while oleic and linoleic dominated the unsaturated fatty acids. It had a ratio of 2.1 of unsaturated to saturated fatty acids. In conclusion, HSA and HSS were significantly different in indexes of peroxide, saponification, and acid. Their contents in terms of density, viscosity, cetane number, refractive index, high heating value, oxidation stability, cold filter plugging point, and fatty acids defined their potential to be used as biodiesel fuel. However, their high acid number can create corrosion problems in engines' fuel delivery channels.

Luque, R. et al. (2012) [46] in their study of advanced biodiesel production, investigated the environmental and economic benefit derived from the production of second generation biodiesel based on advanced processing techniques with the use of

non -edible oleaginous feedstock. The research aimed at providing a general overview of the various processes and techniques suitable for second generation biodiesel production, giving greater attention to the development of an innovative catalyst as well as a concept of a new reactor, as shown in the figure below:

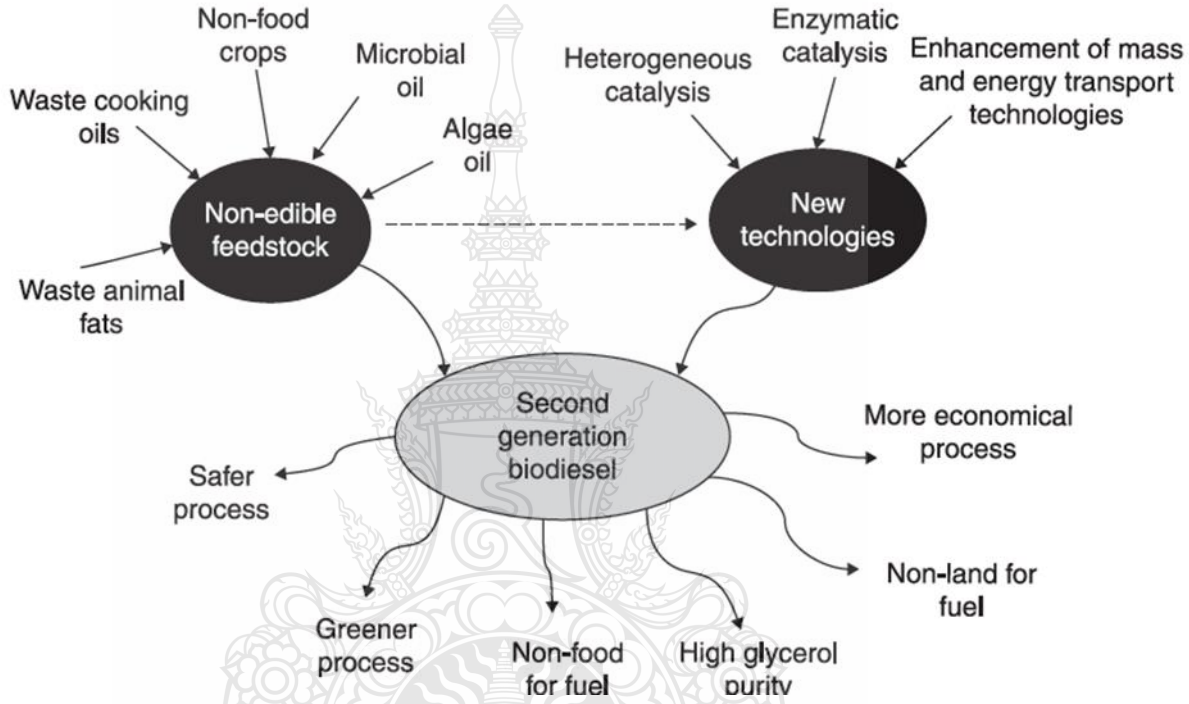


Figure 2.37 Advances in biodiesel production.

In their research, the authors outlined the setbacks of first generation biodiesel production stressing that using a homogeneous alkaline catalyst (NaOH, KOH, NaOMe and KOMe) makes it very imperative to separate the spent catalyst from FAME and the glycerol phase via additional washing steps. Hence it was realized that though biodiesel is a green product, it is not presently manufactured by a green process. They equally added another setback to be the very high sensitivity of the alkaline homogeneous catalyst to free fatty acids (FFAs) and water in the source of oil. FFAs react with alkaline catalyst to form soap, making the glycerol separation process more complicated and cumbersome. Hence, the challenge in the production of biodiesel is the designing of

heterogeneous catalyst. Using a heterogeneous catalyst will result in simpler and cheaper separation processes, lower the effluent load of water and equally reduce capital and cost of energy. With this, fewer inputs will be used, leading to less waste and no soap formation and there will be no need to continually add the catalyst. Moreover, the products will not be neutralized, therefore leading to a higher grade of glycerol that can be produced without any additional purification procedures. The major catalysts used in this study are metal oxides (zirconium, titanium and tin), acid zeolites, sulphonic ion-exchange resin, sulphonated carbon- based and heteropoly acid catalysts. Acid catalysts were found to simultaneously carry out esterification of FFAs and transesterification of triglycerides. However, these catalysts still need important advances or improvements in order to have a positive effect on biodiesel synthesis and production. Such improvements include: increasing the stability of acid sites in order to avoid their leaching, increasing heat stability, avoiding limitations of diffusion, enhancing mass transfer, softer operation conditions and increasing their resistance to water. The researchers found out that biodiesel can also be obtained through a process of enzymatic transesterification, using a catalyst called lipase. The enzyme catalyst is used first without any waste being generated, it is not sensitive to FFAs, has mild rection conditions, and enhances very easy recovery of biodiesel products. This catalyst can be used many times in the process, resulting to a reduction in the energy consumed as well as total wastes produced. However, this enzymatic technique is very expensive, hence difficult to afford. Another non-catalytic technique is the use of supercritical methanol. This takes place in a single homogeneous phase since superficial methanol can be mixed with vegetable oils. The rate of reaction is very fast and the purification process easier. The main disadvantage of this is that it can only thrive in high temperature and pressure rates of between 350 – 400º C and 200 – 400 bars respectively. The microwave technique for transesterification reactions is also noteworthy. This technology has proven to be very efficient in biodiesel production, resulting to high FAME yields in a short time span. The setback of this technique is the scaling -up of the whole process from the level of the laboratory to that of the industry. Also, a major concern about it is its safety. The economic challenges to the biodiesel industry were also identified. They are the high cost of refined vegetable oils, which stands between 70 – 80% of the total

cost of production and the fierce competition of these oils in the food market. Therefore, as a solution to these, they recommended alternative low-cost feedstocks which are readily available in huge quantities. Prominent among these feedstocks is jatropha, which is a potential source of oil for biodiesel production in Asia, Europe and Africa, followed by low-cost animal fat and waste oils as well as oleaginous micro- organisms such as yeast, fungi and bacteria. Conclusively, it was realized that over the past years, biodiesel production had undergone considerable advances, ranging from the conventional base-catalyzed transesterification process using virgin vegetable oils as feedstocks to the advanced processes using non-edible feedstock such as microalgae, oleaginous seeds and micro-organisms as well as waste raw materials such as waste oils and fats.

Jeswani et al. (2012) [47] studied the life cycle sustainability assessment of second-generation biodiesel and how it could substantially reduce the emissions of carbon dioxide from vehicles due to the fact that the bio feedstock used for its production does not contain carbon. The research aimed at examining the life cycle sustainability of second generation biodiesel which are a product of different feedstocks, which are produced in different production systems. The aspect of environmental sustainability such as the usage of water, global warming effects, acidification, eutrophication and the degradation of the ecosystem of biodiesel was also taken into serious consideration as well as the social and economic impacts including the cost of feedstock and capital, and the degree to which the society accept the use of biofuels and their future variability. The researchers pointed out a number of limitations with regards to the use of first generation biodiesel derived from food sources, such limitations include deforestation leading to the unsustainable biodiesel production, water usage and land management (FAO, 2008; IEA, 2010), fierce competition with food production resulting to increased cost of food and food poverty (Bird et al.,2008; Escobar et al., 2009; Fargione et al.,2008; Searchinger et al., 2008). Due to these limitations, a greater attention has been focused instead on the use of non-food sources second generation biodiesel which is free of the limitations of the latter. The sources of second generation biodiesel are essentially non-food such as jatropha, perennial grasses,

short rotation crops (SRC). All of these are energy crops. Other sources include waste biomass especially byproducts from agriculture and forestry, as well as waste cooking oil. Two routes were recommended through which second generation biodiesel can be produced: physiochemical and thermochemical. The physiochemical processing is already well developed unlike the thermochemical, and it strictly involves transesterification of seed oil feedstock of jatropha, castor and waste cooking oil. The thermochemical processing on the other hand is done by gasification followed by fuel synthesis (e.g. Fischer- Tropsch) which is mostly used for feedstock containing lignocellulose. Second generation biodiesel has been found to provide profound benefits which include the possibility to use abandoned lands, reduction of waste products, and the emission of green house gas (GHG) by replacing fossil fuels (IEA, 2010), but this depends on the choice of feedstock used and the cultivation technique. A major advantage of this is that it can trigger job creation, promote rural development and improve the socio-economic condition of low income countries. Presently, second generation biodiesel have been found to have high initial cost of investment and a higher costs for the end- product when compared to fossil fuels and other first generation biodiesel (Eisentraut, 2010). Therefore, the environmental sustainability of biodiesel must be carefully taken into account. The major tool recommended for this was the life cycle assessment (LCA) as shown in the figure below.

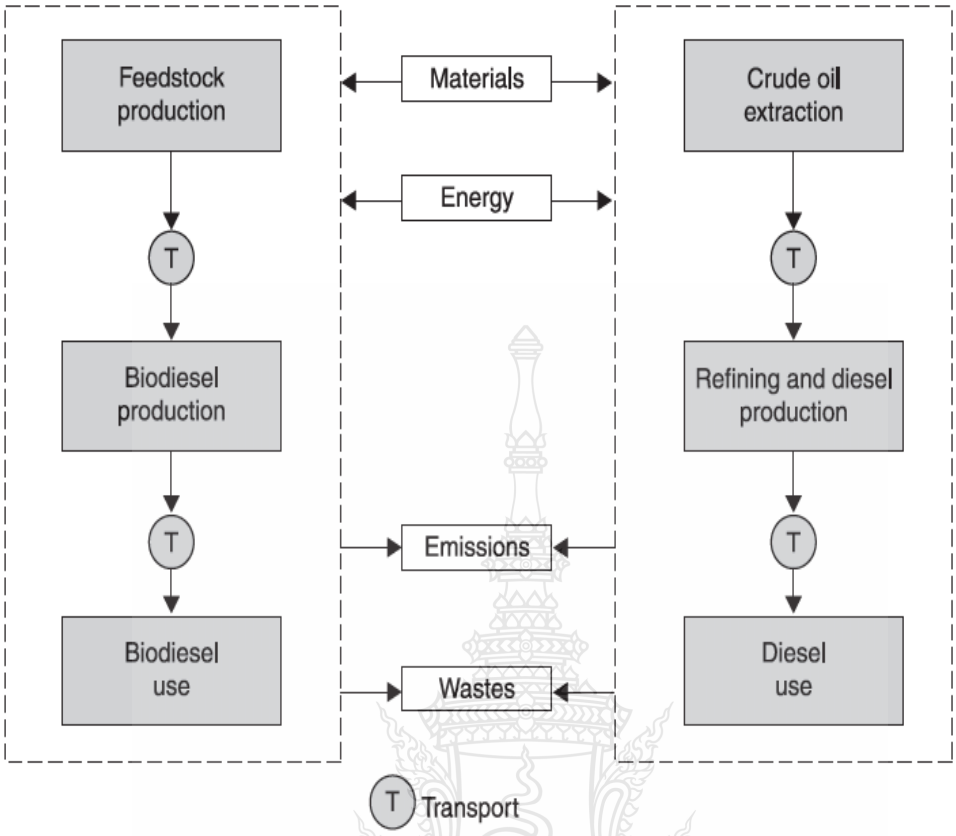


Figure 2.38 Life cycle of biodiesel and fossil diesel from 'cradle to grave.'

The life cycle of biodiesel is a totality of processes ranging from planting, growing and harvesting of biomass, it's transformation to biodiesel right up to when it is used. Each of these life cycle stages poses environmental issues, especially green house gas emission (GHG) depending on the type of feedstock used as well as the procedure of production. The estimation of GHG emissions from second generation biodiesel and net saving as compared to fossil fuel is shown in the table below:

Table 2.47 GHG emissions from second generation biodiesel from different feedstocks.

Feedstock	Origin	GHG emissions (g CO_2 eq MJ^{-1})	Key assumptions	Reference
Miscanthus	Europe	57.9	Allothermal internal circulating fluidised bed (ICFB) gasification	Jungbluth *et al.*, 2007
	EU15	5.4	System expansion credit	Hoefnagles *et al.*, 2010
Switch grass	EU15	11.3	System expansion credit	Hoefnagles *et al.*, 2010
	Various	22–37	System expansion credit; future (2020) technologies	US EPA, 2010
Jatropha	Tanzania	33.4	System expansion credit	Hoefnagles *et al.*, 2010
	India	60	System expansion credit	Reinhardt *et al.*, 2007
	Various	31	Default value[a]; energy allocation	RFA, 2010
SRC/farmed wood	Europe	29.6–57.2	Different process route; centralised entrained flow gasification (cEF); centralised autothermal circulating fluidised bed gasification (CFB) and ICFB	Jungbluth *et al.*, 2007

Waste wood/forest residue	Europe	7.3–13.8	System expansion credit	Edwards *et al.*, 2008
	UK	6	Default value[a]; energy allocation	RFA, 2010
Waste wood/ forest residue	Europe	4–9	System expansion credit	Edwards *et al.*, 2008
	UK	4	Default value[a]; energy allocation	RFA, 2010
Wheat straw	Europe	26.5–64.6	Economic allocation; different process routes: decentralised entrained flow gasification (dEF), cEF and CFB	Jungbluth *et al.*, 2007
Corn residue	US	8.53	System expansion credit; future (2020) technologies	US EPA, 2010
Waste cooking oil	UK	14	Default value[a]; energy allocation	RFA, 2010
	Spain	10.05	System expansion credit	Lechón *et al.*, 2009
	Australia	10.9		Beer *et al.*, 2007
	US	13.3	System expansion credit; future (2020) technologies	US EPA, 2010
	Ireland	20.22	System expansion credit	Thamsiriroj and Murphy, 2011

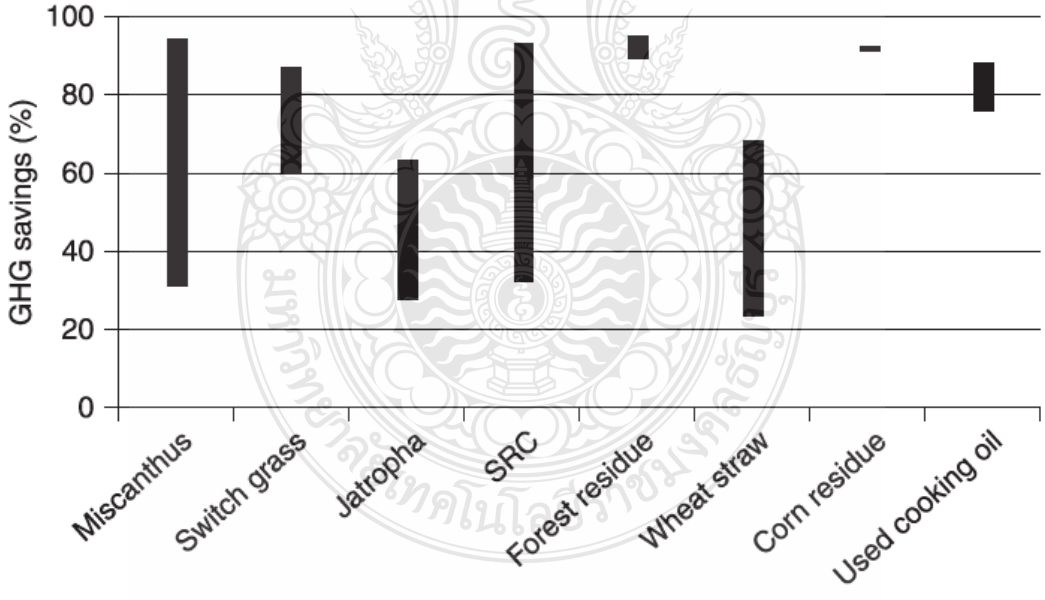


Figure 2.39 GHG savings for biodiesel from different feedstocks and country of origin, compared to fossil diesel.

The potential of biodiesel to cause global warming as a result of the emission of GHG is expressed either in g or kg CO_2 eq MJ^{-1} and the GHG saving from biodiesel compared to fossil diesel are calculated by (EC, 2009) as:

$$GHG_{saving} = \frac{GHG_{fossil\ diesel} - GHG_{biodiesel}}{GHG_{fossil\ diesel}} \times 100\ (\%)$$

The unit of analysis used for comparison is based on the equivalent energy content of the fuels and is usually defined (arbitrarily) as '1MJ of fuel'. The results of both emissions and savings from biodiesel shows that second generation biodiesel has a potential of between 23 – 95% to control GHG emissions over fossil diesel, depending on the type of feedstock used as well as conversion process. In more specific terms, biodiesel produced form waste, more especially forest products and waste cooking oil ensure the highest GHG savings (around 90%), with an average GHG emission of 8 and 13 g CO_2 eq MJ^{-1} respectively (Table 2.47 and Figure 2.39). Comparatively, biodiesel from wheat shows a much lower GHG saving of between 20 – 65%. In conclusion, biodiesel from all energy crops (miscanthus, switch grass, jatropha and SRC) have the potential to control GHG emissions in a range of 28 – 90% over other forms of biodiesel which are essentially first generation biodiesel.

Gad and Hashish (2018) [48] undertook an investigation into the effect of Egyptian roselle biodiesel on the performance and emissions of diesel engines. They ran roselle biodiesel blends with diesel fuel in a diesel engine to examine its effects on performance and exhaust emissions. To achieve this, roselle oil was extracted by mechanical pressing at a 45°C and motor speed of 30 rpm. Then biodiesel was produced by means of transesterification in a conical flask with a hot plate and magnetic stirrer. For the process, roselle oil was preheated to 65◦C and sodium hydroxide (NaOH) which was used as catalyst was dissolved in methanol of 6:1 molar ratio. The reaction time was one and a half hours. Biodiesel was separated from glycerol, dried at 100 °C, and mixed with diesel fuel at varied proportions of ten and twenty percent as in the following table.

Table 2.48 Physical and chemical properties of biodiesel blends B10 and B20 compared to diesel oil using ASTM standards.

Properties	Method	Diesel oil	Roselle Biodiesel (B10)	Roselle Biodiesel (B20)
Density at 15.56°C	ASTM D-4052	835	840	844
Kinematic viscosity, cSt, at 40° C	ASTM D-445	2.5	3.3	5.99
Flashpoint, °C	ASTM D-93	72	85	90
Lower heating value kJ/Kg	ASTM D-224	41670	41017	40866

The density and viscosity of the roselle biodiesel were significantly affected by temperature. An increase in temperature ensued in a decrease in biodiesel density and viscosity (Figure 2.40).

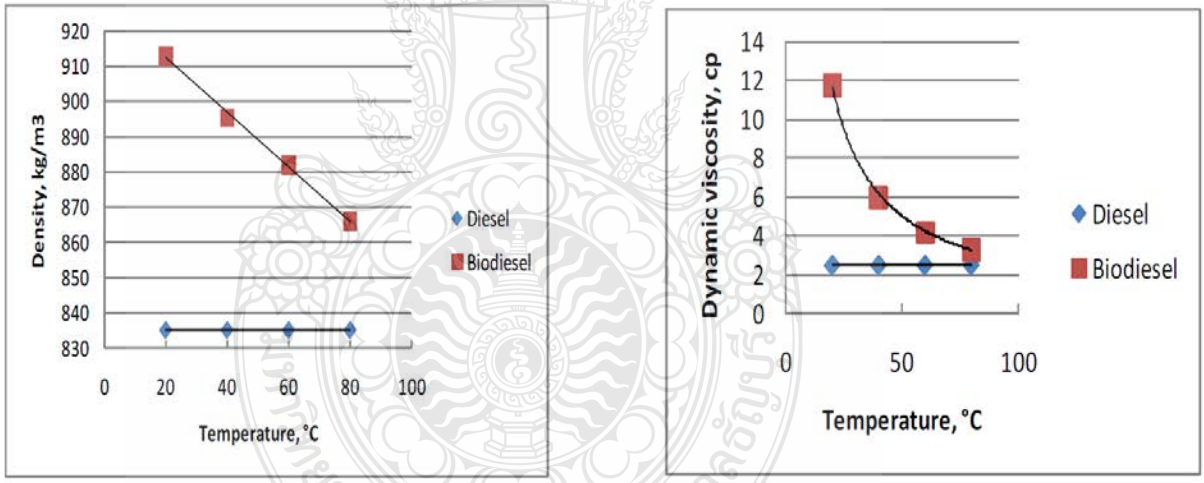


Figure 2.40 Effect of temperature on density and viscosity of biodiesel

Materials used for the experiment included a single-cylinder diesel engine with a maximum power of 5.775 kW at 1500 rpm, an AC generator of 10.5 kW maximum power, an orifice for airflow rate, a manometer for orifice pressure drop, MRU DELTA 1600-V gas analyzer for exhaust emission, and OPA 100 for measurement of smoke opacity. Results from the experiments showed that specific fuel consumptions for roselle methyl ester blends were higher than diesel fuel. Higher biodiesel fuel was consumed by diesel engines than diesel oil at the same power. Roselle biodiesel blends

produced higher thermal efficiencies than diesel fuel due to volatility improvement. Thermal efficiencies of biodiesel blends B20 and B10 achieved maximum increases than diesel fuel by about 4.5 and 10% respectively. With regards to exhaust gas temperature, roselle biodiesel blends have less exhaust gas temperatures at engine loading variation than diesel oil. Since fuel consumption decreases with the biodiesel percentage increase, the air-fuel ratio was found to be higher in biodiesel blends than diesel oil. On mechanical efficiency, biodiesel blends showed more engine output power than diesel fuel due to friction losses. Carbon dioxide (CO_2) emission for biodiesel blends was higher than that of diesel oil because of higher fuel consumption and oxygen content in roselle methyl ester. On the contrary, there was a decrease in carbon monoxide emission for biodiesel blends than diesel fuel. Its high oxygen content enhances improved combustion. Biodiesel blends achieved lower hydrocarbons emissions at all engine loads in comparison to diesel fuel. The hydrocarbons emissions for biodiesel blends increase with engine loads and vice versa. When considering smoke emissions, diesel fuel was higher than biodiesel blends due to its higher oxygen content that causes improved combustion. In conclusion, biodiesel blends showed higher thermal efficiency and lower specific fuel consumption than diesel fuel. Biodiesel blends gave lower emissions of carbon monoxide, hydrocarbons, and smoke but higher emission of carbon dioxide and NO_x. Roselle methyl ester blends up to 20% are used as alternative fuels because performance and emissions were improved compared to diesel fuel.

Gadwal and Naik (2014), [49] engaged in an investigation on hibiscus species seed oils as a potential feedstock for biodiesel production, and its performance in compression ignition engine along with its blends. The study aimed at producing biodiesel from crude hibiscus cannabinus and hibiscus sabdariffa oil through the transesterification process. It analyzed the quality of biodiesel produced from the two hibiscus species and assessed the performance of the produced biodiesel in compression engines using blends. The experimental research underwent various stages beginning from sample collection, processing, and oil extraction from hibiscus cannabinus and hibiscus sabdariffa, then biodiesel production by means of transesterification. The extracted hibiscus cannabinus and H. sabdariffa oils were clear, viscous, yellow in

color, and needed no further refinement. The quantity of the extracted oils was gravimetrically determined and essential properties were checked to ascertain suitability for biodiesel production. The properties were as in the table below.

Table 2.49 Properties of H. cannabinus and H. sabdariffa

Fatty acid composition	***H. cannabinus***	***H. sabdariffa***
(i) Palmitic acid (C16:0)	17.8	18.15
(ii) Stearic acid (C18:0)	5.02	4.09
(iii) Oleic acid (C18:1)	35.60	33.31
iv) Linoleic acid (C18:2)	38.00	38.17
(v) Linolenic acid (C18:3)	2.18	2.09
Density	890.16	919.9
Free fatty acid content	0.664	0.67
Kinematic viscosity	53.00	36.35
Water content	0.058	0.087

Both oils had a high proportion of unsaturated fatty acids especially oleic and linoleic acids, and a lower proportion of saturated fatty acids especially palmitic acid. The average molecular weight of the oil used for the study was 854.1g, and alkali catalyzed transesterification was performed to keep the viscosity at an acceptable level. The free fatty acid of the oils was below one percent and possessed minute traces of water. The fatty acid percentage was found at 0.66% and 0.55% for H. cannabinus and H. sabdariffa respectively. For the transesterification process, 11tr of oil was heated to the temperature of 70-75°C, and methoxide was slowly added. The methoxide which served as catalyst was a mixture of 30 percent methanol and sodium hydroxide. Once the reaction was complete, glycerol was separated, and the biodiesel was washed and dried. 78.8% and 75.5% of biodiesel were produced from 11tr of crude oil of H. cannabinus and H. sabdariffa respectively.

Table 2.50 Yield (%) of biodiesel

S/N	Sample	FFA	Biodiesel before washing	Glycerol (ml)	Biodiesel after washing	Biodiesel after drying (ml)	Yield (%)
1.	H. cannabinus	0.664	790	186	690	670	78.8%
2.	H. sabdariffa	0.652	790	182	685	660	75.5%

The quality parameters of biodiesel produced from H. cannabinus and H. sabdariffa were analyzed. Their density, viscosity, flash point, fire point, and copper corrosion were as follows:

Table 2.51 Quality analysis of Biodiesel

Sample	Density (Kg/m^3)	Viscosity (mPas)	Flashpoint (° C)	Fire point (° C)	Copper corrosion
H. cannabinus	0.875	5.2216	168	178	No corrosion
H. sabdariffa	0.856	4.936	165	175	No corrosion

For the experimental investigation, the CI engine was made to run at a compression ratio of 18:1 and injection pressure of 180 bars with variable loads at constant rated speed. The performance of biodiesel with its blends was as follows. The IP and IMEP

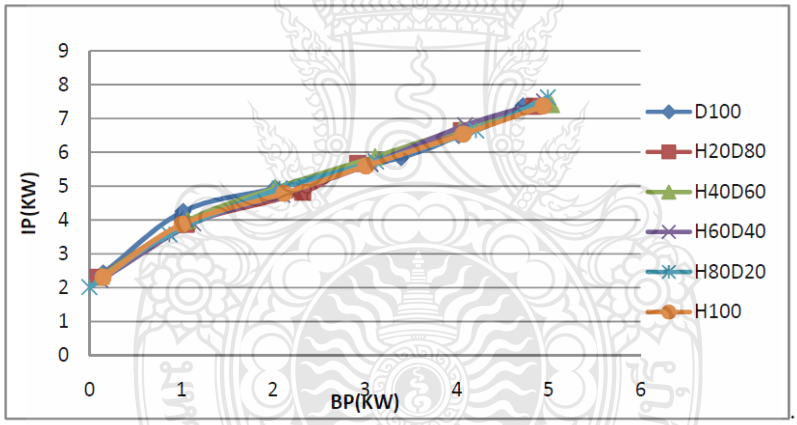


Figure 2.41 Comparison of IP with BP for different biodiesel blend

for H100 were very close to diesel at low load and slightly higher than diesel at higher loads (Figures 2.40 and 2.41). H20 and H40 had higher IThE than diesel and H100 was very close to diesel at a higher load (Figure 2.42). SFC for biodiesel and their blends were higher than diesel (Figure 2.43). H20 and H40 blends were close to diesel at a higher load. H20, H40, and H60 had higher AFR than diesel and H100 was closer to diesel (Figure 2.44). At lower loads, all blends BMEP were closer to diesel, and at higher loads, all blends BMEP values were high (Figure 2.45). H20 and H40 had higher

BThE than diesel and H100 was closer to diesel (Figure 2.46). H20 had higher MechE than diesel and H100 was closer to diesel (Figure 2.47). Finally, the VolE of biodiesel and its blend were lower than neat diesel, compared to H20 which was closer to neat diesel (Figure 2.48).

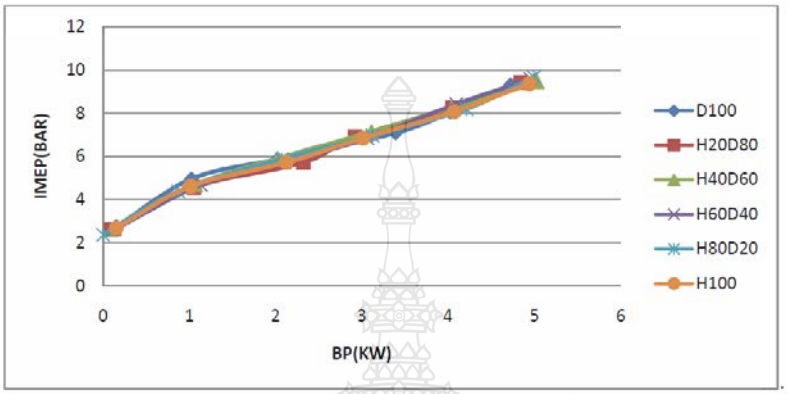


Figure 2.42 Comparison of IMEP with BP for different biodiesel blend

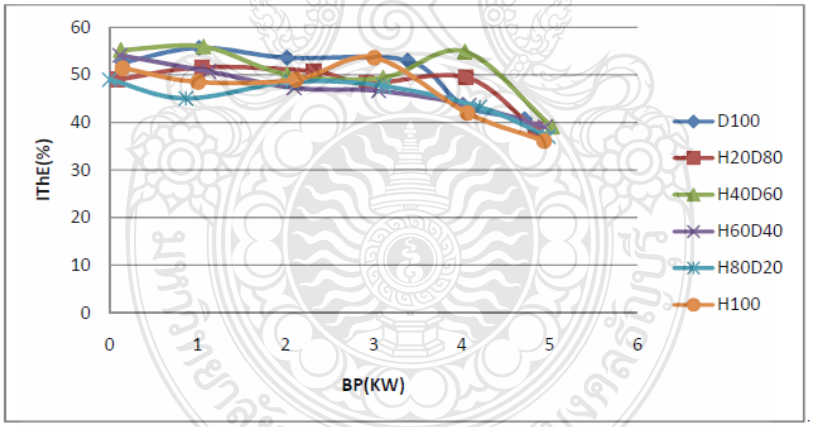


Figure 2.43 Comparison of IThE with BP for different biodiesel blend

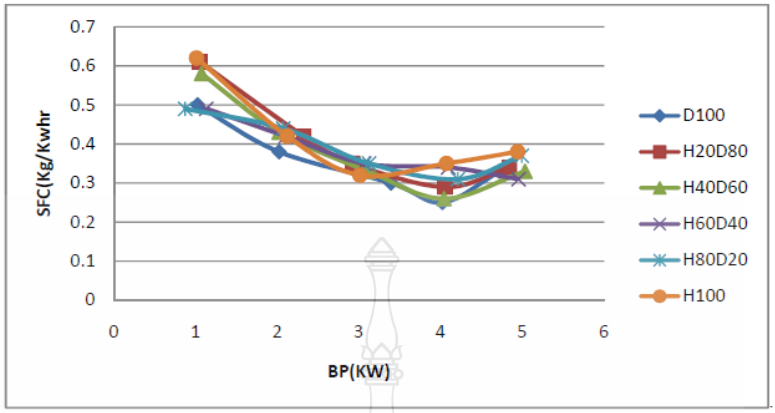


Figure 2.44 Comparison of SFC with BP for different biodiesel blend

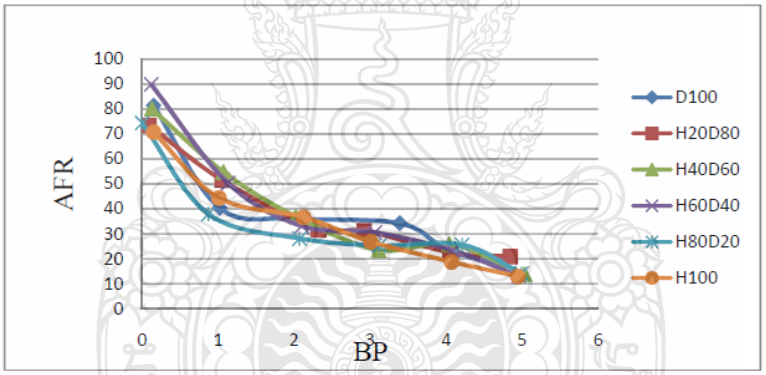


Figure 2.45 Comparison of AFR with BP for different biodiesel blend

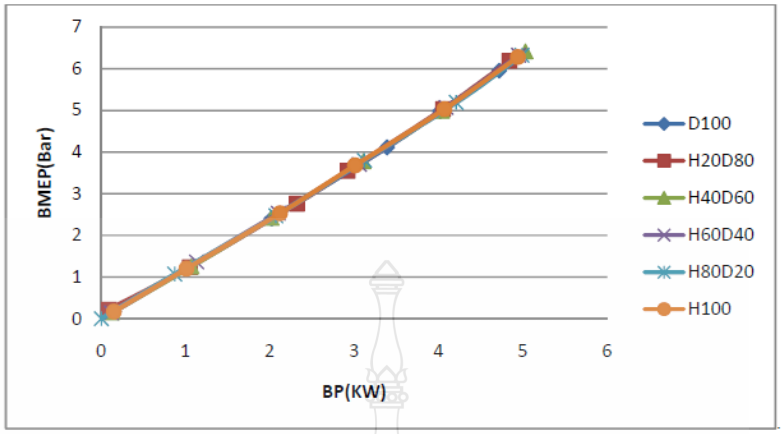


Figure 2.46 Comparison of BMEP with BP for different biodiesel blend

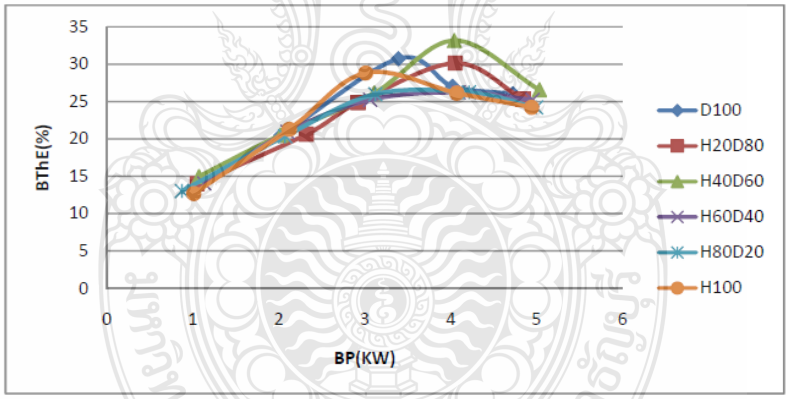


Figure 2.47 Comparison of BThE with BP for different biodiesel blend

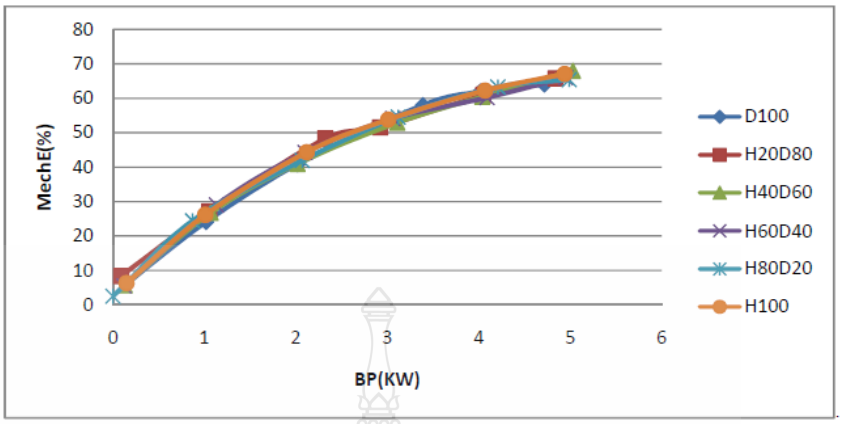


Figure 2.48 Comparison of MechE with Bp for different biodiesel blend

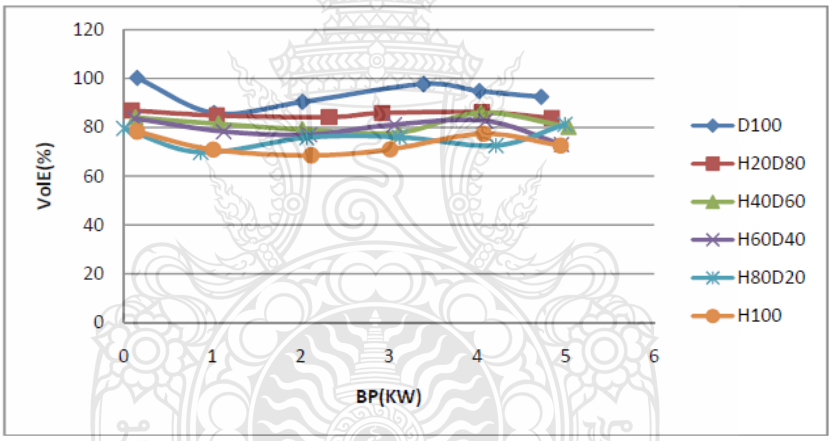


Figure 2.49 Comparison of VolE with Bp for different biodiesel blend

Results from the experiment have shown that biodiesel can be produced from roselle seed oil by alkali catalyzed transesterification with methanol in the presence of a catalyst (NaOH). The study recommended Hibiscus cannabinus and Hibiscus sabdariffa seed oil as supplementary oil feedstocks for biodiesel production.

Dash and Lingfa (2018) [50] undertook a study on an overview of biodiesel production and its utilization in diesel engines. The research aimed at making a general overview of some major and significant works done in relation to the production of biodiesel and its utilization in diesel engines as an alternative source of energy as well

as making a comparative summary on the important developments and recommendations in the biofuel sector. The very first process of biodiesel production identified was the transesterification process invented in 1846 by Rochieder. This process breaks down castor oil for glycerol preparation. Another process is that of Meher et al. which extracts karanja methyl ester from widely available non-edible karanja seed oil. A total yield of more than 90% with a molar ratio 12:1 using potassium hydroxide as a homogeneous catalyst was obtained. When ethyl alcohol was used instead of methanol in the production process, a glycerol separation problem was observed as well as lesser yields. Niak et al. equally developed a two preparation process for karanja biodiesel with FFA of up to 20% by varying oleic acid in the sample of oil. A total yield of 96.6 -97% was obtained with optimum process parameter. Ramadhas et al. on the other hand utilized rubber oil seeds with a low fatty acid of about 17% to produce methyl ester and an optimum 0.5% v/v sulfuric acid with a molar ratio of 6:1in the pretreatment process. In order to ensure maximum yields of esters, the esterified oil must be used for transesterification with 9:1 molar ratio of methanol to oil and 0.5% wt. NaOH. Ghadge et al. also used the same method but a differential aspect was at the pretreatment stage where they employed two steps, intended to reduce the high FFA of mahua oil. It was realized that high amount of methanol was required for a two step pretreatment process.

Table 2.52 Fatty acids profile of popular oilseeds.

Sl. No.	Jatropha oil	Karanja oil	Mahua oil	Rubber oil	Rapeseed oil
Oleic $C_{18:1}$	37.279	49.4	41-51	24.6	64.4
Linoleic $C_{18:2}$	35	19	8.9-13.7	39.6	8.23
Palmitic $C_{16:0}$	14.24	10.6	16-28.2	10.2	3.49
Stearic $C_{18:0}$	6.585	6.8	20-25.1	8.7	0.85
Linolenic $C_{18:3}$	0.086	—	—	16.3	8.23

Table 2.53 Properties of some seed oil

Fuel oil	Flash point (°C)	Viscosity (mm^2/sec)	Pour point (°C)	Calorific value(MJ/Kg)
Diesel fuel	44	1.8	-4	44.637
Jatropha oil	180 - 280	24.5-52.76	-3 to 5	38.2-42.15
Karanja oil	198-263	27.8-56	-3 to 6	34-38.8
Cottonseed oil	210	50	NA	39.6
Rubber seed oil	198	66.2	NA	37.5
Linseed oil	108-242	16.2-36.6	-15 to -4	37.7-39.8
Rapeseed oil	280	39.5	NA	37.6
Amari oil	182	67.7	5	38.829
Pithraj oil	NA	35.093	4	38.729
Mahua oil	232	24.58	15	36

Wang et al. also used a two step catalyzed method for the preparation of methyl from waste cooking oil. In this process, a high acid value of 75.92 mg KOH/gm together with a 38.15% FFA is esterified with a ferric sulfate of 2wt %. This results in a reduction of the value of the acid. Following this, a 1 wt% KOH homogeneous catalyst with conventional transesterification is used for the preparation of biodiesel. Thiruvengadaravi et al., in order to esterify karanja oil, used sulfated zirconia (SZ), and reduced the value of the acid from 12.27mg KOH/g to 1.3 mg KOH/g. It was later transesterified with potassium hydroxide for a period of 2 hours. Hence producing a high quality karanja biodiesel. As biofuels have become the most suitable alternative to petroleum fuels, a major growing concern is their carbon emission tendency. As a result, a lot of considerable effort has been put in to mitigate the carbon emissions of these fuels as well as reduce global warming. Direct and indirect injection diesel engines were used for the performance and evaluation of carbon emissions. Kalam et al. began by using 5% palm oil content and 5% coconut oil and blended it with diesel. With this, they could evaluate the performance and emission potential of an indirect ignition engine. The result was a significant reduction in brake power for both fuel and a corresponding increase in the temperature of the exhaust gas for palm oil and a temperature decrease for coconut blended fuel. It also led to the reduction of CO and HC by a good amount. The emission of NOx equally reduced by 1% for coconut blend and increased by 2% instead for palm blend. Hence drawing the conclusion that due to the more unsaturated fatty acids in palm oil, it causes a comparatively lower emissions

from palm oil blend fuel. Therefore, different feedstock shows different rate of performance and emissions as well operating conditions.

Hasni, K et al (2018) [51] researched the preparation of biodiesel from hibiscus sabdariffa seeds oil using calcium oxide catalyst from waste eggshells. The research aimed at optimizing the transesterification process for the conversion of hibiscus sabdariffa seed oils into biodiesel. The sequence of experiments began with the extraction of hibiscus sabdariffa oil at a constant temperature of 70ºC using hexane as solvent. The nano-catalyst, CaO, was obtained from eggshells, then pre-esterification and transesterification processes were undertaken. Water content was checked with Karl Fischer moisture titrator, kinetic viscosity was tested with Automatic Viscosity Measuring System AKV-201 at 40ºC, Wijs method was employed to measure iodine value, density was assessed with I-type hydrometer, and oxidation stability was determined by Rancimat 743. AOCS Official Method AOCS Cd 3d-63 became the perimeter for defining tocopherol, iodine value, water content, kinematic viscosity, and anacid value. A Mini Pour/Cloud Point Tester MPC-102 was used for pour point and cloud point tests. The cold filter plugging point was checked with Automated Cold Filter Plugging Point Tester AFP-102. Flash point was measured through the aid of e Pensky-Martens Closed Cup Automated Flash Point Tester APM-7. 17.5 wt% hibiscus sabdariffa oil was utilized for the experiment. Its colour was yellowish at room temperature and with a refractive index of 2.16 and an ultrasonic speed of 1.52 ms-1. From the evaluation of its physio-chemical properties, the acid value was 5.486 mg KOH/g depicting the presence of free fatty acids, 2.8 h oxidation stability of the HSO, and 14.228 mm2 kinematic viscosity.

Table 2.54 Physio-chemical properties of Hibiscus sabdariffa seed oil

Properties	Unit	Seed Oil
Oil yield	wt%	17.5
Refractive index	ms-1	2.16
Acid value	mgKOH/g	5.48
Ultrasonic	ppm	1468
Iodine value speed	ms-1	1.52
Oxidative stability	h	2.8
Water content	ppm	55
Kinematic viscosity, 40ºC	Mm^2s^{-1}	14.228

There was a correspondence of FT-IR results to the spectrums of $CaCO_3$ and CaO nano-crystal powders. The spectrums were in the range of 4000-500 nm. $CaCO_3$ had sharp peaks of C-O str. at 714 cm^{-1}, C-O bending at 873 cm^{-1} as well as weak peaks of C-O str. at 1799 cm^{-1} that corresponded to HCO_3 along the peak at 3450 cm^{-1} due to (OH str.) vibrations. The presence of Ca – O bond was attested to by the occurrence of a wide and intense band 1482 cm^{-1} and a weak band at 877 cm^{-1} after calcination at 900ºC. The absorption of -OH groups on the surface of CaO was reflected by the presence of peak at 3657 cm^{-1}. The XRD patterns were archetypical to the nano-crystal with cubic shape. Diffraction peaks which were seen at 32.02, 37.12, 53.80, 64.17, and 67.26 corresponded to the indices 111, 200, 202, 311, and 222 of crystal planes with cubic shapes (B1). Furthermore, the morphological shape of CaO nano-crystals of cubic shape is illustrated by TEM images at 100 nm and 200 nm dimensions.

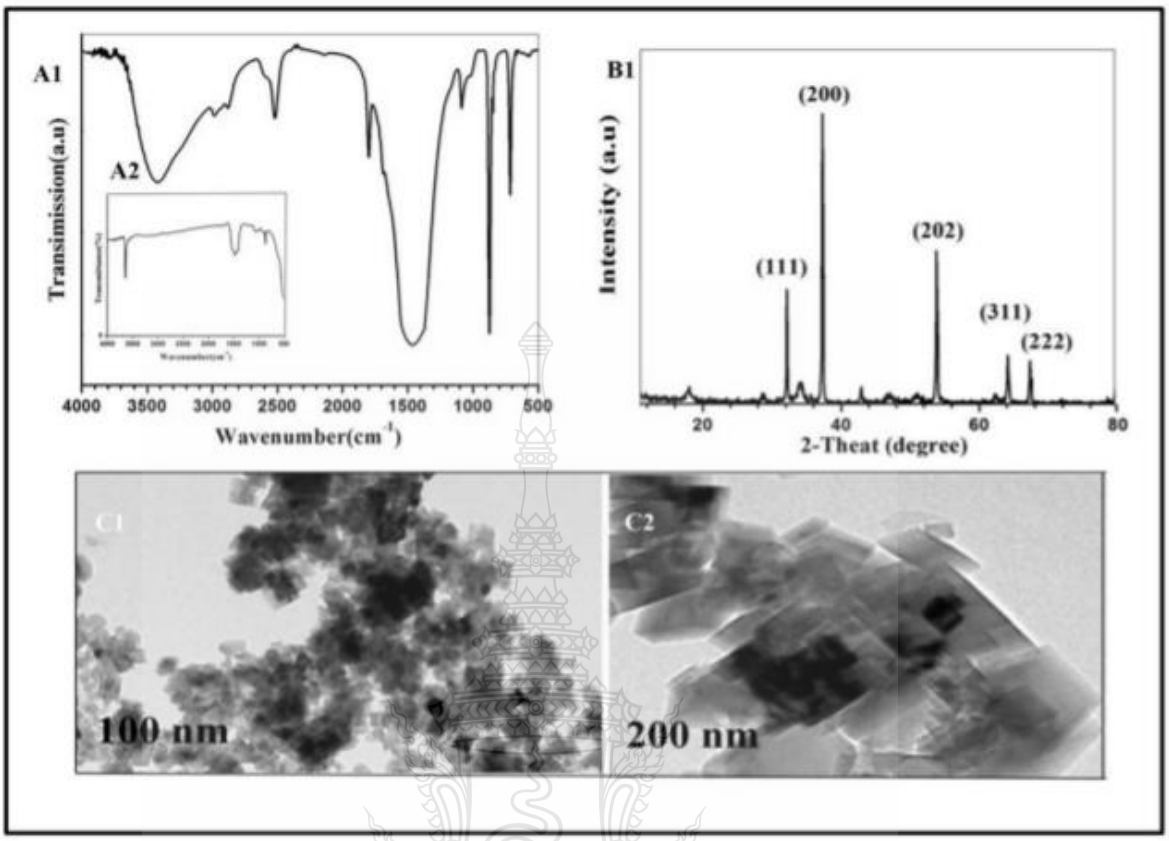


Figure 2.50 A1 and A2 show the FT-IR peaks of $CaCO_3$ and CaO; B1 shows the XRD peaks of CaO; and C1 and C2 show the TEM images of CaO at 100 nm and 200 nm, respectively.

A gas chromatography analysis was conducted on the hibiscus sabdariffa biodiesel to ascertain its fatty acid composition. Methyl palmitate, methyl oleate, methyl stearate, linoleic, methyl arachidate, methyl erucate, and others were predominant fatty acids (Table 2.55). Findings tallied with previous results on Hibiscus sabdariffa methyl esters.

Table 2.55 FAME composition of Hibiscus sabdariffa biodiesel

FAME		**wt%**
Methyl palmitic	16:0	6.57
Methyl steareate	18:0	49.74
Methyl arachidate	20:0	2.21
Methyl oleate	18:1	10.0
Methyl Linoleate	18:2	25.3
Methyl erucate	-	2.94
Others	-	3.24
Total	-	100

The fuel properties were compared according to the international standards – EN 14214 and ASTM D6751. 4.55 mm^2/s of kinetic viscosity was found and that was within the international standards of 1.9-6.0 mm^2/s by ASTM D6751 and 3.5-5.0 mm^2/s by EN 14214. 856 kg/m^3 of HSME density was found. The HSME acid value was measured at 0.054 KOH/g and it falls within the international standards range. Oxidative stability for HSME was seen at 2.47h, the minimum range for ASTM while that of EN 14214 was set at 6 hours. This was far lesser than the 15.2h oxidative stability of diesel fuel. The flash point, cloud point, and cetane number of HSME were found at 161ºC, 3ºC, and 49 min respectively. The physicochemical properties of HSME were within the international standards (Table 2.56).

Table 2.56 Hibiscus sabdariffa (Roselle) biodiesel properties

Plant Seed Oils	Density (mg KOH/g)	Cloud Point (ºC)	Flash Point (ºC)	Oxidative Stability (hour)	Viscosity (mm^2/s)	Cetane Number (min)
Roselle	856	3	161	3.48	4.55	49
Neem	884	14.4	-	7.1	5.21	57.83
Mahua	850	-	208	-	3.98	-
Castor	899	-13.4	-	1.1	15.25	-
Jatropha	880	2.7	135	2.3	4.80	52.31
Karanja	-	-	150	-	4.80	55.84
Malada Pahit	871	2	164	3.0	3.55	51
EN14214	900	-	120	6	5	55
ASTM	-	-3 to 12	170	3	6	47

In conclusion, the conversion of Hibiscus sabdariffa oil into biodiesel was achieved at the optimum operating parameters of 6:1 methanol to oil ratio, the temperature of 67.5ºC, catalyst 1%, and 750 rpm. The achieved yield was 95.01%. Besides the oxidation stability of 3.48 hours, other HSME fuel properties like kinematic viscosity 4.55 mm2/s, acid value 0.027 mg KOH/g, cloud point 3ºC, pour point 1ºC, flash Point 161ºC, cetane index 49 min and density 856 kg/m3 conformed within the range of ASTM D6751 and EN 14214 standard specifications. The results point to the suitability of HSME biodiesel as a diesel fuel alternative and indicate the satisfactory yield through CaO catalyst derived from waste egg shells.

Mabrouk et al (2018) [52] investigated the potentialities of roselle seeds and kernels as sources of oil, protein, and minerals. The study aimed at evaluating the aforementioned seeds as unconventional sources of oil, protein, and minerals. The procedural steps started with extraction oil from roselle seeds and kernel using petroleum ether (40-60 C°) by Soxhlet apparatus for 16h. Fractionation of oil into various classes was undertaken with the aid of a TLC technique. Radwan procedure was adopted in preparing fatty acid methyl esters (FAMEs) of the oil. The FAMEs were separated with the aid of a Shimadzu gas chromatograph (GC4-CM, PFE) at 10% Silar CP on 80/100 chromosorb Q column, FID detector,190-240º C column temperature, 270º C detector temperature, the flow rate of 20ml /min., the gas flow of N2, and chart speed of 5 mm/min. Identification was carried out with standard fatty acid methyl esters, and a triangular method was used to measure areas under each peak then each fatty acid was expressed in percentage. Through the use of Abbe refractometer (Leica Mark II NARP 79190), the refractive index (RI) of the oil sample was checked according to the AOAC. The colour of the oil was measured with Hunter System, and its viscosity was checked with Brookfield digital viscometer at 40°C. After oil extraction, the roselle seed meal and roselle kernel meal underwent protein analysis according to the AOAC method. Supernatant was obtained from samples of roselle seed meal and roselle kernel meal dissolved in phosphate buffer pH 8, shook for an hour at room temperature, and centrifugated at 5000 xg for 10 min. Through the discontinuous buffer system, the sodium dodecyl sulphate (SDS-PAGE) technique was carried out. A 10% slab gel was prepared. Electrophoresis separation was performed using Mini-PROTEANII (Bio-Rad) at 75 V through the stacking gel followed by 125 V to the end of electrophoresis (2 hr). Staining and destaining were performed in line with Hames and Rickwood methodology. Beckman Amino Acid Analyzer Model 119 CL was employed to ascertain amino acid composition. Minerals: Ca, Mg, Fe, Cu and Zn were measured by atomic absorption spectrophotometric (Perkin-Elmer Instrument Model 2380), K by flame photometer, and P was calorimetrically measured at 630 nm.

From the experiment, Roselle seed oil was found to contain 8 lipid fractions: polar lipids (phospholipids), monoacylglycerols, 1,2 and 2,3 diacylglycerols, sterol, 1,3 diacylglycerols, free fatty acids, triacylglycerols, and hydrocarbons and sterol esters. With regards to fatty acid composition, the result indicated that roselle oil was composed of 30.31 % saturated fatty acid especially palmitic (22.44%) and stearic (5.77%) acids, and 69.7% unsaturated fatty acid especially linoleic (37.61%) and oleic (31.27%). Unsaturated fatty acid had a greater proportion than saturated fatty acid at a ratio of 2.3:1 (Table 2.57).

Table 2.57 Fatty acid composition of crude RS oil (% of total fatty acid content).

Fatty acids	**Value**
Lauric C12:0	0.64
Myristic C14:0	0.71
Pentadecanoic C15:0	0.32
Pentadecnoic C15:1	0.11
Palmitic C16:0	22.44
Palmitoleic C16:1	0.6
Heptadecanoic C17:0	0.32
Stearic C18:0	5.77
Oleic C18:1	31.27
Linoleic C18:2	37.61
Linolenoic C18:3	0.11
Arachidic C20:0	0.11
Total saturated fatty acids (S)	**30.31**
Total unsaturated fatty acids (U)	**69.7**
S/U ratio	**1:2.3**

The physicochemical characteristics of roselle seed oil included yellowish colour at room temperature, 0.9197 specific gravity at 25ºC, 30 cp viscosity value at 40ºC, iodine value of 99.47 g of I2/100 g, and peroxide value of 7.52 meq O_2 /kg.

Findings reveal that the composition and properties of roselle oil were similar to other edible vegetable oils such as cotton seed oil and corn oil. This widens the expanse of its industrial applications and value. On roselle proteins, there was a significant difference

Table 2.58 Physicochemical properties of crude roselle seed oil

Property	*Value
(at 18ºC)Refractive index	0.15 ± 1.472
at 25ºC Specific Gravity	0.12± 0.9197
(at 40ºC (cp Viscosity	30.00 0.5 ±
: Colour	
*a	0.94
*b	9.94
*L	29.85
(g 100 / Iodine value (g of I_2	0.7 ± 99.47
(kg oil / meq O_2) Peroxide value	0.27± 7.52
Saponification value	0.3± 194.4
(oleic acid %) Free fatty acids	0.03 ± 0.83

Mean ± standard deviation (SD)*.
Each value represents the average of three determinations.

between proteins of seed and kernel in their number of bands and intensity (Table 2.59).

The amino acid composition as indicated by the result was basically dominated by aspartic, glutamic, arginine, and aromatic amino acids. Its existent proportion of isoleucine, total aromatic amino acids, threonine, valine, and histidine was greater than FAO reference values, while leucine, lysine, and total sulfur amino acids were contrastingly lower than FAO reference values. Inferentially, roselle seed could be taken as a good source of high protein.

Table 2.59 Amino acid profile of RS and RK produced using different soaking conditions (g/100 g protein).

Amino acid	RS	RK				FAO* Pattern
		A	B	C	D	
Isoleucine	4.09	5.29	5.46	5.55	5.13	2.80
Leucine	6.28	6.35	5.67	7.58	8.57	6.60
Lysine	5.35	4.61	5.23	3.48	3.4	5.80
Methionine	0.95	1.51	1.52	1.63	2.43	-----
Cystine	0.11	0.31	0.37	0.17	0.15	----
Total sulphur amino acids	1.06	1.82	1.89	1.80	2.58	2.50
Tyrosine	3.17	4.23	4.28	3.89	3.51	----
Phenylalanine.	4.98	5.79	6.36	6.21	5.97	-----
Total aromatic amino acids	8.15	10.02	10.54	10.10	9.48	6.30
Threonine	3.76	3.65	2.55	2.02	2.11	3.40
Tryptophan	ND	ND	ND	ND	ND	1.10
Valine	6.16	6.86	6.70	6.82	6.22	3.50
Histidine	4.62	3.38	3.14	2.63	2.61	1.90
Total essential amino acids	39.47	41.98	41.18	39.98	40.1	33.9
Arginine	10.18	12.48	12.97	73.12	12.75	
Aspartic	11.66	8.71	8.97	93.8	8.11	
Glutamic	18.19	19.10	19.79	20.38	20.55	
Serine	4.61	2.64	2.36	2.07	1.53	
Proline	2.40	3.37	3.21	3.13	3.55	
Glycine	6.30	4.52	4.43	5.36	5.7	
Alanine	4.49	4.69	4.84	5.06	5.21	
Total non-essential amino acids	57.83	55.41	56.57	57.66	57.4	
Total amino acids	97.30	97.49	97.75	97.64	97.50	

ND= not determined. *FAO pattern: FAO/WHO/UNU (1985).

RS =Roselle seed. RK = Roselle kernel.

A°25 =C/10 hr. B= 45°C/2 hr. C= 65°C/1 hr .D= 75°C/15 min.

The results indicated that the mineral composition of roselle seed flour was predominantly potassium (1145 mg/100 g), phosphorus (482 mg/100 g), magnesium (242 mg/100g), and calcium (239 mg/100 g) while iron, copper, and zinc were had lower concentrations. Dehlling process caused a variety of effects on the mineral content.

Table 2.60 Mineral composition of RSF and RKF produced using different soaking conditions.

*(mg/100g sample)Mineral	RSF	RKF			
		A	B	C	D
K	1145	1030	950	938	973
Mg	242	245	246	248	244
P	482	532	528	554	544
Ca	239	230	233	233	241
Fe	9.7	9.5	9.8	9.8	9.6
Cu	10.3	22.6	20.1	12.4	19.1
Zn	9.8	12.3	13.1	10.9	10.7

*On dry weight basis.

In conclusion, Mabrouk et al (2018) [52] submitted that roselle seed has good nutritional quality. It is an auspicious source of protein fortification for many foods and a potential food ingredient. Roselle seed oil could be used as edible oil supplements and for food industrial applications. In addition, roselle seed as a whole or its flour after oil extraction is a potent source of basic minerals especially potassium, phosphorus, magnesium, and calcium.

Ahmed et al (2020) [53] examined alkali-catalyzed transesterification of hibiscus sabdariffa seed oil for biodiesel production. The study aimed at extracting biodiesel from roselle seed oil and investigating its quality. For the experiment, roselle seeds were collected, washed, heat-dried, and milled to proper size. Oil was extracted from 100 g of roselle seed powder with the use of Soxhlet and n-hexane as solvent. The extracted oil was subjected to physiochemical analysis where colour was visually observed at room temperature, density was checked with a density meter, and the refractive index was measured with a pen Refractometer at room temperature. The oil's acid value was gotten through the direct titration method, free fatty acids titrated according to AOCS Official Method Ca 5a-40, and peroxide value analyzed in line with AOAC Official Method 993.20. Then transesterification of crude roselle oil using methanol and potassium hydroxide as catalyst; with a ratio of oil to alcohol 1:8 at 65°C. The quality of produced biodiesel was investigated and compared to international standards. The fatty acid composition of the produced biodiesel was determined by GC-

MS. Results of the experiment had it that a 12.65% yield of oil was obtained. The oil was yellowish in colour and with an acid value of 11 mg KOH/g, 1.467 ms^{-1} refractive index at 25°C, free fatty acid of 5.5%, saponification value of 252 mg KOH/g, 0.915 g/mL density, and ester value of 241 mg KOH/g (Table 2.61).

Table 2.61 Physiochemical properties of Roselle seed oil

Property	Units	Value
Density	g/mL	0.915
Refractive Index (40°C)	ms^{-1}	1.467
Acid value	mgKOH/g	11
Free Fatty acid	%	5.5
Saponification	mgKOH/g	252
Peroxide value	meq/kg	2
Ester value	mgKOH/g	241

The transesterification process produced a high biodiesel yield of 96% with 0.80 g/mL density and 44.63 API, 0.742 of kinematics viscosity at 40˚C, pour point at less than 51˚C, and 0.65% of Micro Carbon Residual (MCR). These were within the range of ASTM D6751 and EN 14214 standard specifications.

Table 2.62 Properties of biodiesel produced from Roselle seed oil

Test name	Test method (ASTM)	Unit	Result
Density@ 15˚C	D4052	g/mL	0.80261
API	D4052		44.63
Pour point	D97	˚C	<-51
MCR	D4530	%wt	0.65
Kinematics viscosity @ 40˚C	D445	CSt	0.742

API: American Petroleum Institute Gravity
MCR: Micro Carbon Residue

The biodiesel was subjected to GC/MS analysis which unraveled the dominant properties to include linoleic acid, elaidic acid, and palmitic acid. The components of the produced biodiesel and the GC-MS spectrum were as below.

Table 2.63 Components of biodiesel produced from Roselle seed oil

Fatty Acid	Systematic name	Formula	Area%
Myristic acid	Tetradecanoic	$C_{14}H_{28}O_2$	0.21
Lycopodic acid	11-Hexadecenoic acid	$C_{16}H_{30}O_2$	0.23
Palmitic acid	Hexadecanoic acid,	$C_{16}H_{32}O_2$	17.8
Malvalic acid	2-octylcyclopropene-1-heptanoic acid	$C_{18}H_{32}O_2$	0.42
Linoleic acid	9,12-Octadecadienoic acid	$C_{18}H_{32}O_2$	33.0
Elaidic acid	9-Octadecenoic acid	$C_{18}H_{34}O_2$	29.8
Oleic acid	9-Octadecenoic acid(z)	$C_{18}H_{34}O_2$	0.46
Linoleic acid	10,13-Octadecadienoic acid	$C_{18}H_{32}O_2$	3.54
Linoleic acid	10,13-Octadecadienoic acid	$C_{19}H_{34}O_2$	3.17
α-Linoleic acid	9,12,15-octadecatrienoic acid	$C_{19}H_{32}O_2$	0.14
α-eleostearic acid	9.cis,11.trans.t,13.trans-Octadecatrienoic	$C_{18}H_{30}O_2$	1.95

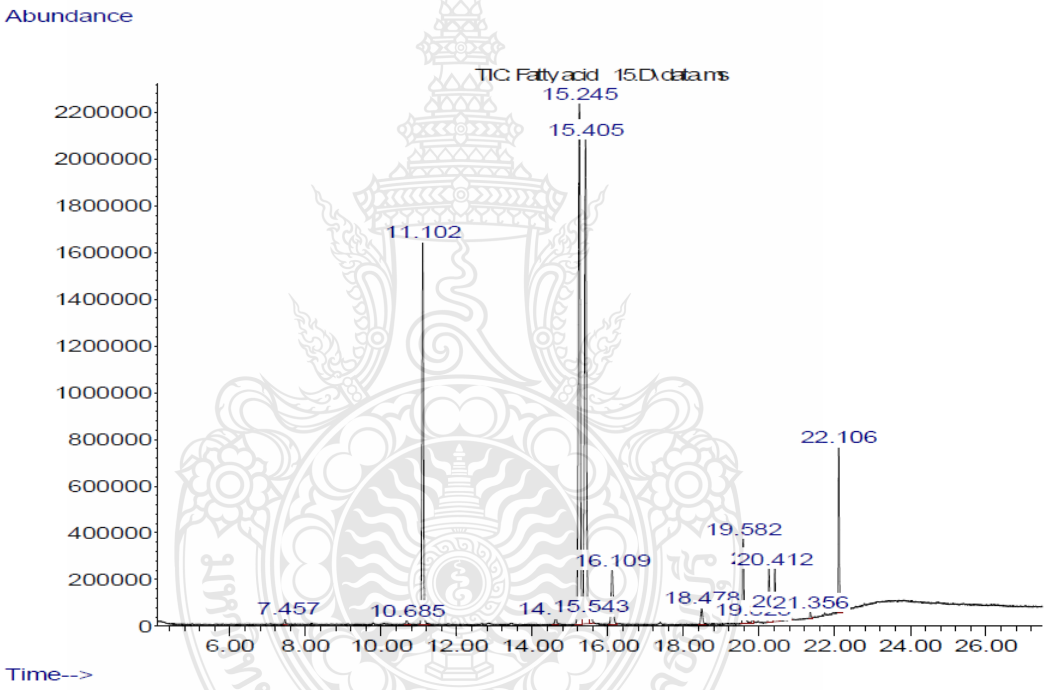


Figure 2.51 GC-MS spectrum of biofuels produced from Roselle

Inferentially, the study indicated the suitability of biodiesel from roselle oil for diesel engines and its satisfactory yield due to potassium hydroxide. It recommends the use of biodiesel on CI engines because of its lower emission of carbon monoxide. Methanol which is needed for transesterification is not costly and possesses short chain molar size. However, particular cognizance should be given empirically ideal molar ratios to avoid excesses of methanol.

Navas et al. (2020), [54] carried out the study of a sustainable process for biodiesel production using Zn/ Mg oxidic species as active, selective and reusable heterogeneous catalysts. The aim of the study was to described the preparation and characteristics of MgO and ZnO as pure and mixed catalysts with the support of γ-Al_2O_3. The performance of these catalysts was studied in the transesterification of soybean oil and castor oil with methane and butanol in a bid to produce biodiesel. They used the following to ascertain the characteristics of the prepared catalyst; XRD (X-ray) diffraction, SEM – EDS (scanning electron microscopy – energy dispersive X-ray spectroscopy), CO_2- as well as N_2- adsorption. For the experimental research on the one hand to ascertain the catalyst preparation, Mg and Zn catalysts pure and mixed in different proportions were used. They were prepared using the conventional method of coprecipitation of carbonates proceeded by calcination (Lee et al. 2013). The original catalysts were supported on γ-Al_2O_3, then meshed and sieved to 60 -100 mesh. The total amount of supported oxides was 0.17mol (MgO + ZnO) for each 100g of alumina. The Mg/Zn catalyst mixtures were with an atomic ratio of 0.5, 1.0, 1,5 and 2.0. For the preparation, a substantial amount of Mg and Zn nitrates were dissolved in an aqueous suspension of γ-Al_2O_3. Then, $(NH_4)_2CO_3$ 1 M was added and stirred strongly. The pH was controlled at a value of 9, using NH_4OH. The solution was stirred for 2 hours. After that the solid was filtered and dried at 60 °C overnight. The active phases were obtained after calcination at 500 °C. On the other hand, the determination of the composition elements of the catalysts was achieved by atomic absorption spectroscopy using a Varian 240 equipment. Digestion was carried out for each of the samples using concentrated HCI on a hot plate. The lines employed were 202.6 nm for Mg lamp, and 213.9 nm for Zn lamp. At a temperature of -196º C, the texture of the different catalysts was determined by N_2 physisorption. The crystalline phases present were determined using an X-ray diffractometer Philips PW 1740 (Cu Kα radiation, $\lambda = 0.154$ nm). The samples were scanned from 5° to 75° at the scanning speed of 1 min^{-1}. To determine the amount of base in the catalyst, a thermogravimeter was used to quantify the amount of

CO_2 absorbed. The FTIR spectra of the fresh and post reaction 0.5 Zn/ Mg catalyst were recorded in the diffuse reflectance mode on a Thermo Avatar 360 instrument using a DTDs detector. The average spectra from 120 scans ranged between 400–4000 cm^{-1} at a resolution of 4 cm^{-1}. An oxidation thermogravimetric analysis was performed using a thermobalance (Shimadzu TGA -50) in order to possibly detect the presence of carbonaceous deposits on the post reaction catalyst. This was done in a temperature of 10° C/min and an air/He feed (2:1) and a mass of 10 mg for both fresh and post – reaction catalyst. The temperature range and amount of weight loss were recorded as a function of time. To ascertain the catalyst performance, it was done in the transesterification process, using soybeans oil and castor oil separately without mixing them and two alcohols, that is methanol (Circarelli, 99.8%) and butanol (Merck, 99.4%) separately without mixing them. This was in order to facilitate the differentiation of the oil, in terms of their characteristics and composition as shown in the table below:

Table 2.64 Typical fatty acid composition (%) of soybeans and castor oil (Meneghetti et al. 2006, 2007)

Fatty acid	Fatty acid content (%)	
	Soybean oil	Castor oil
Myristic (14:0)	0.2	–
Palmitic (16:0)	16.0	1.8
Stearic (18:0)	2.4	–
Oleic (18:1)	23.5	–
Linoleic (18:2)	51.2	11.2
Linolenic (18:3)	8.5	–
Ricinoleic (18:0(OH))	–	87.0
Total C18	85.6	98.2
Free fatty acids (FFA)	0.1	1.2

The experiment was conducted in a three-necked batch glass reactor with a dimension of 250-cm^3 , with a reflux condenser and mechanical stirrer installed in it. The alcohol/ oil molar ratio, the amount of catalyst and the reaction temperature were all considered at 6:1; 5 wt%, and 60° C for10° C methanol and 80° C for butanol (Navas et al. 2018). A thorough analysis of the product was carried out by a GC (gas chromatography) method according to EN (European Standard) 14105 and ASTM (American Society of Testing Materials) D6584 [EN14105; ASTM D6584], using a GC-2010 Plus Tracera

Gas Chromatograph, which was equipped with a BID detector. A MEGA-Biodiesel 105 (15 m × 0.32 mm × 0.10 μm) capillary column was used. Then samples were taken after 2, 4 and 6 hours of reaction. The pre-injection treatment of the samples, as well as the chromatographic conditions, is detailed in Navas et al. 2018.

Table 2.65 Textural properties and chemical composition of the prepared catalysts.

Catalyst	S_{BET} (m^2/g)	V_{pore} (cm^3/g)	d_{pore} (Å)		MgO (wt%)[a]	MgO (wt%)[b]	ZnO (wt%)[a]	ZnO (wt%)[b]	Zn/Mg[c]	Zn/Mg[d]
			Mesopores	Macropores						
MgO/γ-Al_2O_3	223	0.43	32	57	6.85	nd	–	–	–	–
0.5 Zn/Mg	168	0.32	36	57	4.67	4.14	4.61	5.72	0.5	0.68
1 Zn/Mg	232	0.35	37	51	3.42	3.81	6.26	5.47	1.0	0.71
1.5 Zn/Mg	266	0.41	37	52	2.74	3.15	8.30	9.21	1.5	1.45
2 Zn/Mg	182	0.39	37	51	2.28	1.82	9.22	6.22	2.0	1.69
ZnO/γ-Al_2O_3	173	0.42	32	67	–	–	13.83	nd	–	–
γ-Al_2O_3	216	0.45	nd	75	–	–	–	–	–	–

From an observation of the experimental results, it can be seen that the experimental atomic ratio was quite similar to the theoretical one, for all the sample catalysts, except for the 2 Zn/Mg catalyst in which the zinc content is substantially lower than expected. The difference found may be accounted for, being due to the pH control in the preparation, since MgO and ZnO require different pH values for an optimal precipitation (Ngamcharussrivichai et al. 2008; Lee et al. 2011). For the textural properties of the different sample catalyst, it was found that the pore distribution showed the presence of macro and mesoporous with differential diameter. The mesoporous diameter for pure oxides catalysts were slightly lower than those of Zn/Mg catalysts. Observing the macropore values, ZnO/γ-Al_2O_3 catalyst presented a value of 67 A, a considerably higher value than that observed for MgO/γ-Al_2O_3 catalyst and the Zn/Mg mixtures, that range between 52 and 57 Å.

Shrivastava et al (2021) [55] investigated the performance and emission characteristics of a compression ignition engine fueled with roselle and Karanja biodiesel. The research aimed to examine the technical possibility of using roselle and

Karanja biodiesel as an alternative fuel in the CI engine. It further analyzed the effects of these biodiesels by changing the compression ratio and engine loads. To achieve these, roselle and Karanja seeds were collected, oils were extracted, and filtered to avoid impurities. The transesterification process was undertaken with the aid of methanol as an alcoholic agent, and sodium hydroxide and potassium hydroxide were used as catalysts. A single-step transesterification technique was adopted for the production of biodiesel fuel from roselle oil and Karanja oil. The same procedural steps were taken on both oils. A comparative analysis of the physicochemical properties of diesel (B0), roselle biodiesel (LA100), and Karanja biodiesel (KB100) showed some similarities and differences. They were as in the table below.

Table 2.66 Physicochemical Properties of Diesel, Roselle Biodiesel, and Karanja Biodiesel.

Fuel	B0	LA100	KB100	Unit
Density	830–840	878	880–913	kg/m^3
Viscosity at 40°C	2.5–3.11	5.64	3.99–5.71	mm^2/s
Heating value	42.2	38.72	38.91–42.13	MJ/kg
Cetane number	48	52	52	—

The fatty acid composition of roselle and Karanja oil were also checked. This is because fatty acid plays an essential role in engine performance. Biodiesel with lower fatty acid composition enhances better engine performance.

Table 2.67 Fatty Acid Composition of Roselle and Karanja Oil.

Fatty Acid Composition (%)	Palmitic Acid	Oleic Acid	Stearic Acid	Linolenic Acid	Linoleic Acid
Roselle oil (*Hibiscus sabdariffa*)	18.0	33.6	4.06	2.02	38.05
Karanja oil (*Pongamia pinnata*)	11.55	51.62	7.40	2.62	16.54

Results from the experiment showed that brake thermal efficiency gradually increases with an increase in engine load, and vice versa. At higher engine loads, heat loss reduces which in turn increases engine power. On a general note, improved thermal efficiency of the engine is recorded whenever there is an increased combustion ratio. A comparative look at the brake thermal efficiency of all tested fuels at full load condition was recorded at 31.6%, 32.4%, and 33.3% for Karanja biodiesel; 31.2%, 32.1%, and

32.8% for roselle fuel; and 32.6%, 33.1%, and 33.7% for diesel fuel at 16.5, 17.5 and 18.5 combustion ratio. The brake specific fuel consumption for diesel, roselle biodiesel, and Karanja biodiesel decreases with an increase in combustion ratio. BSFC for the biodiesels appeared to be higher at all combustion ratios. At CR 16.5, 17.5 and 18.5, Karanja biodiesel exhibited BSFC of about 0.2, 0.283, and 0.278 kg/kW-h respectively; roselle biodiesel exhibited 0.298, 0.289, and 0.283 kg/kW-h respectively; and diesel fuel exhibited 0.263, 0.256, and 0.248 kg/kW-h respectively. Observing from the data so far, an increase in combustion ratio was more for biodiesel fuel than diesel. With regards to exhaust gas temperature, it increases with an increase in engine load for all tested fuel. For KB100 (Karanja biodiesel), the exhaust gas temperature was observed to be about 300.72ºC at 16.5 CR load, 309.6ºC at 17.5 CR, and 317.22ºC at 18.5 CR; for LA100 (roselle biodiesel), 294.17ºC was observed at 16.5 CR, 304.4ºC at 17.5CR, and 313.37ºC at 18.5 CR; then in diesel fuel, 319.16ºC was seen at 16.5 CR, 324.59ºC at 17.5 CR, and 338.75ºC at 18.5 CR. Comparatively, diesel fuel exhibited higher exhaust gas temperature than biodiesels. Considering the emission of carbon dioxide, there was a reduction in smoke emission with the increase in combustion ratio. KB100 emitted 843.96, 822.8, and 812.28 g/kWh; LA100 emitted 858.6, 838.6, and 824.5 g/kWh; and diesel fuel emitted 828.9, 812.28, and 800.93 g/kWh at full load condition and combustion ratios of 16.5, 17.5, and 18.5. In conclusion, the performance and emission characteristics of roselle and Karanja biodiesels were comparatively investigated on changing combustion ratios of 16.5, 17.5, and 18.5 and engine loads of 50% and 100%. There was a respective 4.53% and 4.87% increase in brake thermal efficiency of roselle and Karanja biodiesels with a concomitant increase in combustion ratio from 16.5 to 18.5. The brake specific fuel consumption was reduced by 4.31% for Karanja biodiesel, 5.30% for roselle biodiesel, and 5.70% for diesel fuel as the combustion ratio increased from 16.5 to 18.5. There was a decrease in smoke emission by 2.63% for KB100, 5.30% for LA100, and 5.70% for diesel fuel as the combustion ratio moved from 16.5 to 18.5 and at full load condition. An increase in combustion ratio from 16.5 to 18.5 ensued in a concomitant increase in exhaust gas temperature and nitrogen oxide, and decrease carbon dioxide emission. Better performance and emission characteristics of compression ignition engine fueled with roselle and Karanja

biodiesels were recorded at higher combustion results. The experimental result, therefore, proved that roselle and Karanja biodiesel could be potent alternative fuels for a diesel engine.

Delvi et al (2019) [56] conducted research on biodiesel production utilizing diverse sources, oils' classification and esters, performance, and emission characteristics. The objective of the study was to undertake a detailed assessment of the various aspects of biodiesel engineering. These aspects consisted of biodiesel feed stocks, a variety of methods adopted in biodiesel production such as pyrolysis, microemulsion, dilution, and transesterification. The aim extended to include understanding the effect of biodiesel blend magnitude on the performance of engine parameters such as brake power (BP), brake thermal efficiency (BTE), and fuel properties like cloud point, flash point, calorific value, kinematic viscosity, density, and cetane number as well as the economic viability, emission characteristics and Greenhouse gas emissions. The main feed stocks of biodiesel that were edible oils were extracted from soybeans (glycine max), safflower, rice bran oil (oryza sativa), barley, sesame (sesamum indicum l.), groundnut, sorghum, wheat, corn, coconut, canola, African oil palm (elaeisguineensis), sunflower (Helianthus Annuus), rapeseed (Brassica napus L.). Common non edible oils that serve as sources of biodiesel included extracts from cotton seed (Gossypium hirsutum), jatropha curcas, Pongamia (pongamiapinnata), Pongamia (camelina sativa), Karanja or honge (pongamiapinnata), cumaru, Cynara cardunculus, mahua (madhucaindica), coffee ground (Coffea arabica), abutilon muticum, neem (Azadirachta Indica),nagchampa (calophylluminophyllum), passion seed (passiflora edulis), salmon oil, tall (Carnegiea gigantean), croton megalocarpus, pachira glabra, aleuritesmoluccana, Terminalia belerica jojoba (Simmondsia Chinensis), rubber seed tree (hevcabrasiliensis). Known sources of animal fats that are feed stocks for biodiesel consisted of fish oil, beef tallow, chicken fat poultry fat, pork lard. Other sources include algae (cyanobacteria), bacteria, terpenes, poplar, switchgrass, fungi, miscanthus, latexes, microalgae (chlorella vulgaris).

A cursory look at the engine performance parameter vis-à-vis biodiesel showed varying values according to feed stocks. Overall, the kinematic viscosity of biodiesel

was ten to fifteen times higher than diesel fuel due to its bigger atomic mass and chemical structure. The most extreme permissible limit concurring to ASTM D445 ranges were (1.9–6.0 mm^2/s) and (3.5–5.0 mm^2/s). Density was measured at the temperature run of 15 to 20ºC concurring with EN ISO 3675/12185 and ASTM D1298 20ºC. Biodiesel exhibited exceptionally high flashpoint that is close to 150ºC than diesel fuel with 55-66ºC. Cloud point and flashpoint were measured according to ASTM D2500, EN ISO 23015, and D97 procedures. Cetane number was higher in biodiesel than conventional diesel. The chemical composition of biodiesel makes it more vulnerable to oxidative degradation than fossil diesel fuel. when comparing the brake power of engines operating on pure plant oils or blends with diesel, they varied 10 to -18 %. Due to high viscosity and low volatility, the brake thermal efficiency of biodiesel was low. Brake specific fuel consumption of plant oil was similar to that of fossil diesel fuel.

Table 2.68 Physiochemical properties of universally used feed stocks

Oil or Fats	Density 15 °C (kg/m^3)	Kinetic viscosity at 40°C (mm^2/s)	Cetane No (°C)	Flash Point (°C)	High heating value (MJ/kg)	Cloud point (°C)	Calorific value MJ/kg	Pour point (°C)	Iodine No (°C)	Acid (neutralization) value (mg KOH/g)
Canola	–	4.42	37.6	160	39.7	-3.3	–	-9	–	0.01
Soybean	913.8	4.039	37.9	254	39.6	0.9	39.76	–	128-143	0.266
Sunflower	880	4.439	49	160	39.6	3.4	–	–	–	0.027
Palm	864.42	4.5	54.6	135	–	16	–	15	54	0.24
Peanut	848	4.42	53.59	166	39.8	0	40.1	-8	67.45	0.28
Safflower	885.5	5.8	56	148	–	-5	38.122	–	–	–
Mesua	898	6.2	54	112	39.5	–	42.23	3	–	0.01
Rice Bran	872	4.811	51.6	430	–	–	41.38	269	–	0.48
Maclura	889	4.66	48	180	39.4	-5	–	-9	125	0.4
Cotton seeds	875.7	4.09	51.43	150	39.4	7	40.43	6	–	0.16
Jatropha	879.5	4.8	51.6	135	38.65	2.7	39.23	2	104	0.4
Neem	868	5.213	–	76	–	9	39.81	2	–	0.649
Karanja	931	6.13	55	95	–	7	43.42	3	–	0.42
Mahua	874	3.98	65	208	36.0	–	36.8	6	–	0.41
Linseed	874	3.752	52	160	39.3	-3.8	–	-15	–	0.058
Coconut	807.3	2.726	–	114.8	–	–	–	–	–	0.106
Rapeseed	882	4.43	54.4	170	37.4	-3.3	37	-12	–	–
Tobacco	888.5	4.23	51.6	165.4	–	–	–	–	136	0.3
Beef tallow	–	4.624	–	–	–	–	–	–	–	0.147
Roselle	880.1	4.588	–	130	–	–	–	-1	62	0.43
Okra	876	4.01	55.2	156	–	1.10	–	2.12	–	0.38
Rubber	–	5.81	–	130	–	4	36.5	-8	–	–
Coffee	–	4.852	–	160	–	0.2	–	–	–	0.076
Diesel	850	1.3-4.1	40-55	60-80	42	-20	42-46	-35	38.3	0.062
Calophyllum Inaphyllum	888.6	7.72	51.9	151	–	38	–	–	85	0.76

The prevalent biodiesel production methodologies were transesterification, pyrolysis, micro-emulsion, and dilution. With regards to emission, biodiesel was considered a carbon-neutral product. It had reduced emission of carbon monoxide, nitrogen oxide, and unburned hydrocarbons as compared to diesel fuel. Sequel to this, it was observed that biodiesel had more capacity to reduce greenhouse emissions than other fossil fuels. It was also observed that the output power and mechanical efficiency of CI engines using biodiesel were low but with high fuel consumption, contrasting diesel fuel.

Khan et al. (2020) [57] investigated the performance and emission analysis of high purity biodiesel blends in diesel engines. The research focused on the production of a much purer biodiesel with a high cetane index of 61.4 using the electrolytic separation and emulsification process. The main feedstock used was a mixture of vegetable oil, giving a maximum production yield of 84%. The experiment made use of materials and chemicals such as vegetable oil, methanol, potassium hydroxide, distilled water, and sodium chloride. In the experimental procedure, 0.5 M solution of potassium hydroxide (KOH) is made in methanol and added to vegetable oil in the ratio 1:5. Forty-seven grams of powdered KOH were dissolved in 2 L of 97% pure methanol and added to 10 L of vegetable oil. With continuous stirring, the mixture is heated to 60°C and the temperature is maintained for 1 hour. The mixture is then allowed to cool and settle for 24–36 hours. This settling time ensures that the transesterification is complete and glycerol separates out. The electrolytic separation method is used to enhance the speed and efficiency of glycerol. Here, a metal plate is connected to AC terminals, then dipped into the mixture. The speed of separation is directly proportional to the voltage while the purity of biodiesel depends on the time. At the end, after the glycerol settles, it is removed from the bottom. It was found out that if glycerol is present in biodiesel, it will result to damaging the storage tank of the fuel and the fuel filter due to unconventional combustion in the engine. In the step that follows, biodiesel is washed to remove potassium hydroxide and other impurities, then distilled water is added and stirred vigorously, resulting to the formation of an emulsified solution. Initially, the required amount of distilled water is 2 liters. The emulsion is then separated by addition of hot sodium chloride solutions and then the mixture allowed to separate. After this, layers will form with pure biodiesel settling on the top while sodium chloride, potassium

hydroxide and the soapy solution stay at the bottom. The obtained biodiesel is then dried at a constant temperature of 100º C for a period of 1 hour, in order to completely get rid of any residual water and unsaturated methanol. The diesel product was tested according to various standards including ASTM D93 for flash point, ASTM D97 for pour point, ASTM D240 for calorific value, and ASTM D86 for distillation. The results can be seen in the table 2.69

Table 2.69 Comparison of biodiesel and diesel properties

	Biodiesel (B100)	Diesel	Units
Density	940.2	813.6	$kg\,m^{-3}$
Cloud point	−4.6	−2	°C
Flash point	58	62	°C
Pour point	−5.9	−6	°C
Calorific value	41.63	45.80	$MJ\,kg^{-1}$
CCI	61.4	40.9	–

The experimental engine is setup on a test bed, in order to ascertain the performance and emission parameters for clean diesel and biodiesel blends. Both blends were compared experimentally. The technical specifications of the test engine as well as the experimental engine mounted on a test bed are shown in the table and figure below:

Table 2.70 Engine specification and Equipment.

Make	Perkins AD3.152 diesel
Bore	91 mm
Stroke	127 mm
Engine type	Liquid cooled in-line three cylinder
Maximum power	46 hp/34.3 kW
Maximum torque	172 Nm
Compression ratio	16.5:1
Rated rpm	2250
Engine capacity	152 ci/2.49 L
Starter volts	12 V
Maximum efficiency	29.2%
Coolant capacity	9.8 L
Air cleaner	Dual dry element
Dynamometer	Eddy current dynamometer
AVL 437C smoke meter	Filter smoke number (FSN)
AVL 444-DI gas analyzer	HC, CO, CO_2, O_2, and NOx

Figure 2.52 Engine mounted on a test bed.

The blend samples used in the experiment all had different properties. The sample values were first calculated theoretically and then later confirmed experimentally before

using them to calculate and determine the results. In total, there are 4 blends; B0, B5, B20 and B50. Blend B5 is used to test the degree of responsiveness of the engine, if biodiesel is used as a fuel enhancer not a replacement. This blend also has thermophysical properties similar to those of diesel. The B20 blend is chosen as a comparative fuel percentage. The B50 blend is chosen to test the response of the engine if biodiesel in the later stages is used as a replacement. Moreover, B50 is fuel mixture where the thermophysical properties of the blend are an average of diesel and biodiesel. The biodiesel blend properties are shown in the table below:

Table 2.71 Biodiesel blend properties.

Blend	Percentage of biodiesel (% by volume)	Density ($kg\,m^{-3}$)	Calorific value ($MJ\,kg^{-1}$)
B0	0	813.60	45.80
B5	5	819.93	45.59
B20	20	838.92	44.96
B50	50	876.90	43.72

The results show that the biodiesel produced from the described method above were purer and cleaner than other methods in which similar samples were used. The biodiesel produced has a significantly high calculated cetane index (CCI) of 61.4. A high cetane value suggests a lower percentage of unsaturation, as studied by Gopinath et al. This further signifies an improved oxidation stability. Moreover, the blends of this biodiesel will tend to have reduced delay in the engine ignition in accordance with the findings of Killol et al. However, the density of biodiesel is found to be 40 kg/m^3 higher than the standard limit of 900 kg/ m^3 while the flash point is significantly lower than the minimum standard limit mainly due to the presence of methanol in the biodiesel. Furthermore, during the distillation of the biodiesel, it was so efficient such that, it was found that 95% of the volume was recovered leaving behind very small amounts of wastes which could possibly consist mainly of long chain hydrocarbons and aromatic compounds. The fact that most of the biodiesel was recovered as the distillation stage indicates that it had very low amounts of impurities. Hence, the biodiesel produced from

this method is found to be of good quality. Very small amounts of methanol present in the biodiesel would assist combustion inside the engine. Conclusively, the blend B5 is found to be the most effective in the situation of maintaining the engine performance and improving the engine emissions. The power and torque output of B5 are similar to diesel while there is a significant decrease in BSFC accompanied with an increase in BTE at engine speeds higher than 1800 rpm. This means that B5 is an excellent fuel enhancer additive and as such should be recommended for high speed operations. The maximum increase in BTE is found to be 1.75% which 6% more than diesel at 2250 rpm for B5. B5 also gives significantly improved emissions for CO and unburnt HCs.

CHAPTER 3

RESEARCH METHODOLOGY

This chapter describes the determination of Seed Moisture, Oil Extraction, Investigation of the Properties, Bio-oil characterization, Gas Chromatography – Flame Ionization Detector, Bomb Calorimeter production of biodiesel from the Roselle seeds coming from agricultural waste material and useless to return the benefits of renewable fuels as well as test features of the product to be compared to international standards ASTM and DIN.

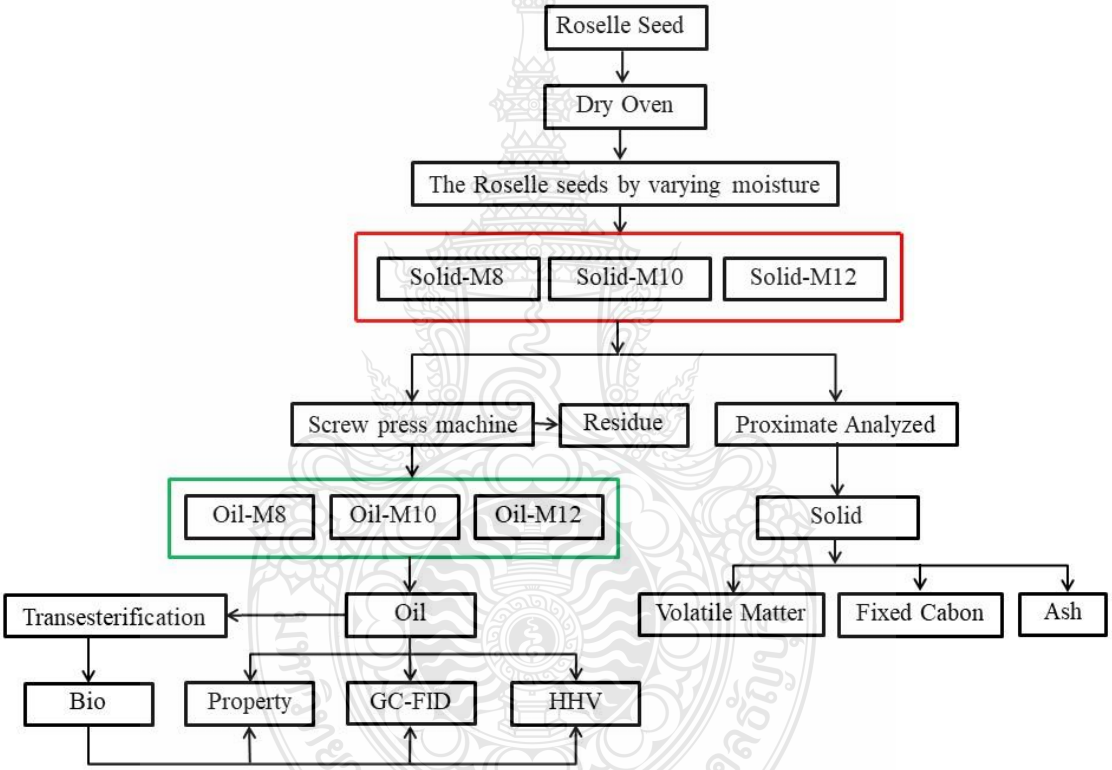


Figure 3.1 Research organization chart

3.1 Raw Material

The roselle seeds are brought from Amphoe Doembang Nangbuat, Changwat Suphanburi in the central region of Thailand. Information regarding the seed was provided by the District Agricultural Office in Doembang Nangbuat. The cultivated plantation area of Roselle is 2,400,000 m^2 with productivity of 12,000 tons/year. The harvested Roselle was sun-dried for 7-9 hours and then cleaned on a vibrator grille

device that is the Forced Convection Oven – model FD53 to removed the moisture**.** The Roselle oil was obtained using a machine screw extractor**.** Subsequent property investigations that were tested in compliance with ASTM and EN standards for the organic solution, ash and carbon by using a Simultaneous Thermal Analysis, Netzsch model STA 449 F3 *Jupiter*®.

Figure 3.2 Showing the process of raw materials Roselle seeds.

3.2 Determination of Seed Moisture

From Figure 3.3, the method and equipment used to ascertain the moisture content, by standards specified by the International Seed Testing Association (ISTA), used a Forced Convection Oven – model FD53; the container size of which is 100mm in diameter and a 10mm high dehumidifier with silica gel, machine grinding, sieve size of 0.5mm and a machine scale resolution of 0.0001g. The Roselle seed was ground in the grinder using a sieve to filter out the resultant product to 0.5mm size. First, the weight of the empty container used for weighing the raw (unprocessed) material was taken and the result was saved. Next, the ground Roselle seed was placed in the container and weighed before drying, and the results were saved. The ground Roselle seed was dried

at a temperature of 130°C for an hour then cooled in a suction cup for 30-45 minutes. After drying, the resultant product was weighed and the results were saved. The process was repeated six times and the results were saved for calculation of the average percentage of moisture. Repetition of the results was performed to give greater accuracy in the data calculation. The formula used is shown below:

$$\text{Percent Moisture} = (V_2\text{-}V_1/V_2\text{-}V_3)\times 100 \quad (1)$$

Where: V_1 = Weight of empty container (g), V_2 = Weight of empty container and raw material before drying (g), V_3 = Weight of empty container and raw material after drying (g), V_2-V_3 = Moisture loss, and V_2-V_1 = Weight of raw material (g)

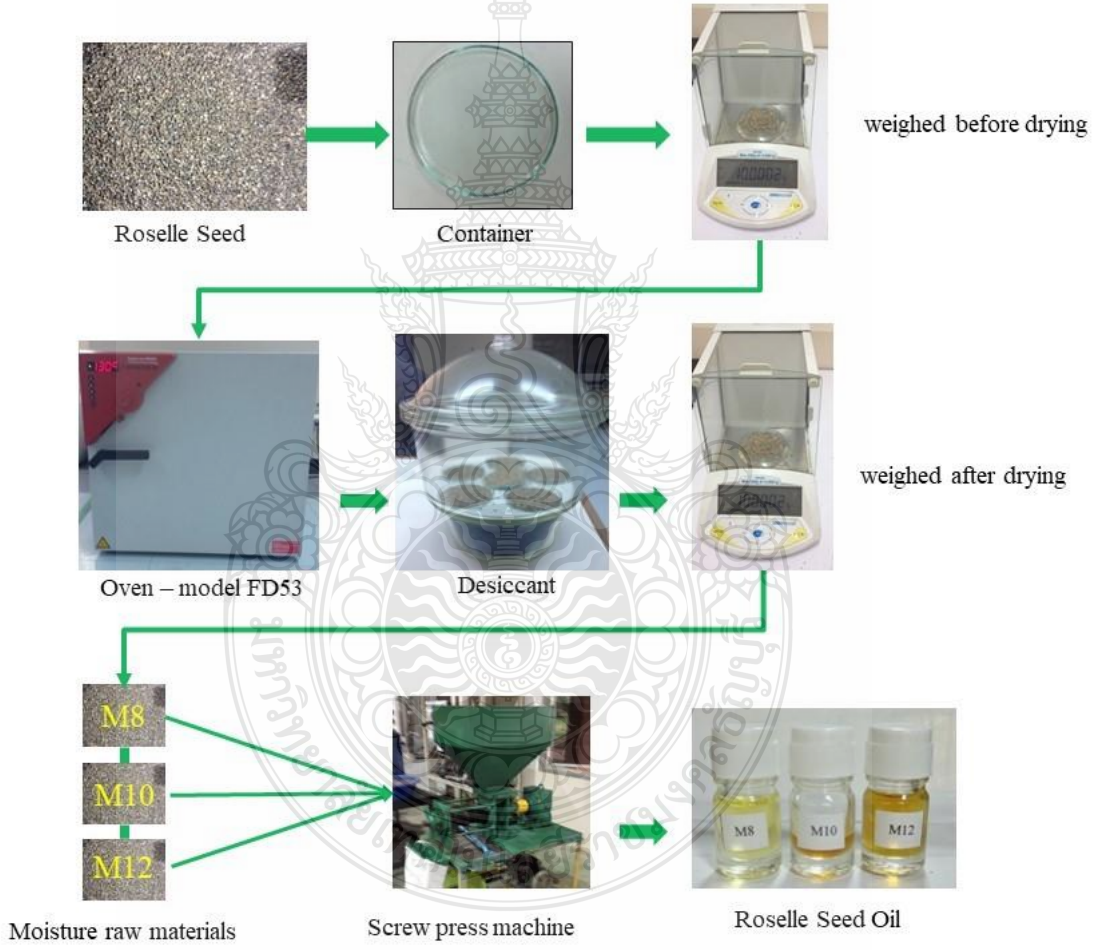


Figure 3.3 Procedures for Determination of Seed Moisture.

3.3 Oil Extraction

From the Figure 3.4, Roselle seeds with three different derived moisture contents of 8 % (M8), 10 % (M10), and 12% (M12) were tested. These different moisture contents were placed in a cylindrical container with a volume 0.0468 m^3. Oil

from the seed was extracted using a Screw Extractor powered by a three-horsepower motor, which forced the fluid through a circular grooved steel plate placed on a belt in front of the motor with a diameter of 102 mm, and then a second steel plate, 203 mm in diameter. The motor gear ratio was 1:40 with a transmission screw placed at a speed of 19 rpm. The seed oil under compression passes from the screw plate through the filters with a filter paper of 1 2 5 mm in diameter. The volume of oil remaining can then be compared.

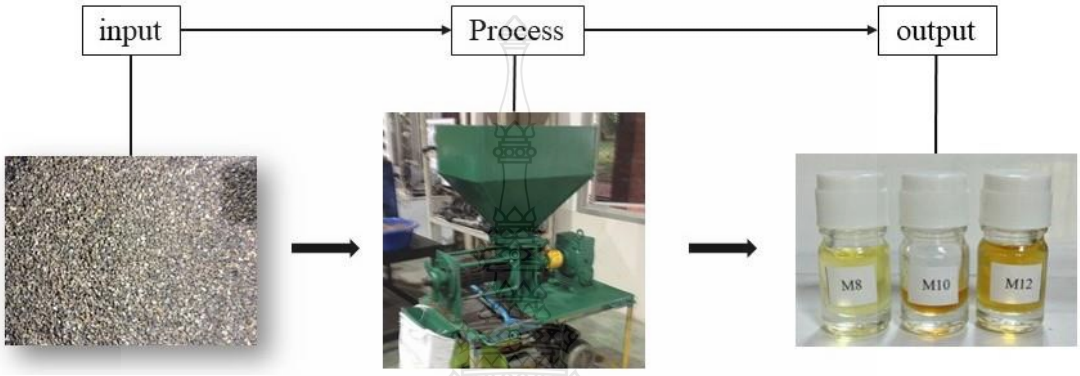


Figure 3.4 Procedures for Oil Extraction.

Figure 3.5 Sample of oil filtration

3.4 Proximate Analysis

The Proximate Analysis methodology was used to analyze the Roselle seed oil through a Netzsch model STA 449 F3 *Jupiter*® analyzer. The Roselle seed was ground to 0.3 mm and a volume of 150mg for the three moistures of M8, M10, and M12 each was placed in containers of either platinum or aluminum. A test sample was used to

compare the temperature of the substance with a reference sample. The containerized samples were heated and the weight change was measured using a weighing machine. In addition, a sample was measured by suction for the evolved heat of the sample by measuring the weight using a thermocouple and temperature gauge. Burning the raw material and experimental samples under different conditions were used to find the relationship between volatile matter, ash, and fixed carbon. The analyzed results are shown in graph and table form.

3.5 Investigation of the Properties

The following properties were investigated: acid value, viscosity, Iodine value, and flashpoint. The methods used were as follows. The acid value was examined by the ASTM D664 – 17, a standard testing method for the acidic value of petroleum products by potentiometric titration. To bring the Roselle seed oil and alcohol PH value to a middle volume of 50mm, 2-3 drops of phenolphthalein were mixed by titration with a solution of sodium hydroxide 0.5N. As each drop was titrated into the container of oil, the mixture was shaken until it changed to pink. The volume of sodium hydroxide used to complete the reaction was then used to calculate the acid value of the oil. To find the experimental viscosity value outlined in ASTM D445, a Viscometer was used. The defined volume of Roselle seed oil was put into a test tube, which was placed in a jar of water to maintain a controlled temperature at 40ºC. The time taken for the Roselle seed oil to flow through the strainer multiplied by a constant of the test tube gave the kinematic viscosity. Using the Pearson titration method, and following the European guidelines DIN EN14111, to determine the Iodine value of the Roselle seed oil, a prepared mixed compound sample was titrated with a solution of trisulphate 0.1N to find the Iodine value. The Flash Point according to the ASTM D93 standard was determined using Pensky Martens Closed Cup equipment. Roselle seed oil was poured into the cup to the prescribed level and placed on the base support of the machine. A thermometer was placed in the cup, which was then covered and a motorized spinning blade was switched on to stir the mixture. A gas hose was connected to the flame system. The flame was pointed at the stirred volatile compound, and by twisting a mechanism on the lid of the cup, the flame could dip in the channel of the test cup. The flame was then returned to its normal position. To stop the flame, the motor was stopped. The measured temperature of the flammable substance was thus the value of the flashpoint.

3.6 Bio-oil characterization

Gas Chromatography – Flame Ionization Detector

Figure 3.6 shows the chemical composition of bio-oil produced from Roselle seed analyzed qualitatively by Gas Chromatography – Flame Ionization Detector (GC-FID). and a DB-23 capillary column (60 m × 0.25 mm ID × 0.25 μm). GC oven program: Initial Temperature: 50º C, Hold Time: 1 min. Rate 1: 25º C/min to 175º C, Hold Time: 0 min. Rate 2: 3º C/min to 220º C, Hold Time: 3 min. Rate 3: 2º C/min to

230ºC, Hold Time: 5 min. Post Run:240º C, 2 min, run time: 34 min, Inlet temperature: 240ºC, Mode: Constant pressure: 33.00 psi, Nominal initial flow: 2.1 mL/min, Split mode: Split 5:1, Injection volume: 1 µl

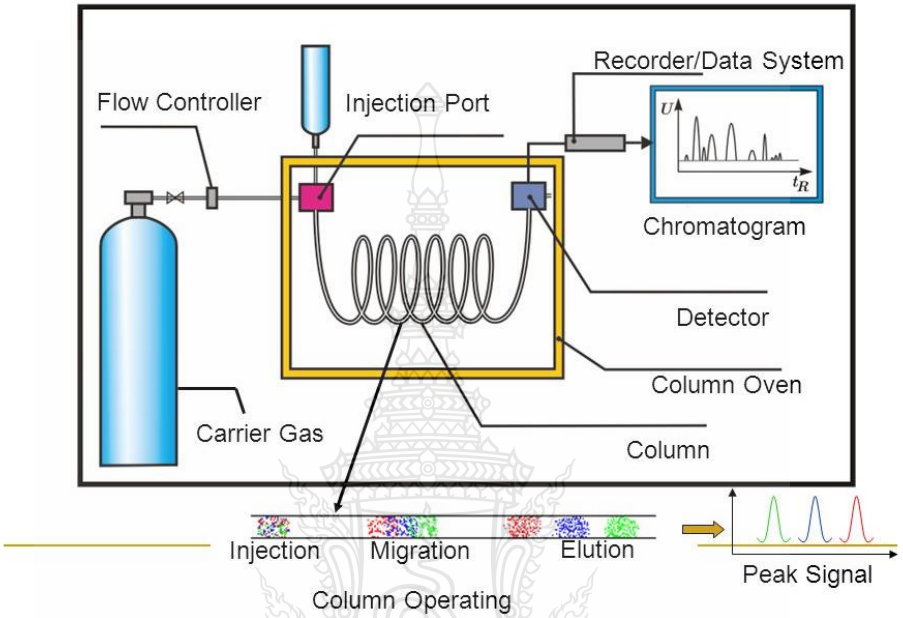


Figure 3.6 Gas Chromatograph sample

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Bomb Calorimeter

For analysis of the amount of heat from combustion reactions with pure water, as heat energy is received, it causes the water temperature to rise. It can be calculated as heat value in the heat energy range 1000-10,000 cal/g for solid raw materials ground into powder and the liquid must have a minimum energy value of 1000 cal/g.

3.7 Titration method

As shown in Figure 3.7, bring sodium hydroxide of 50 g and mix with distilled water of 500 ml. The result will be 10% alkaline solution. Then bring an alkaline solution of 5 ml and mix with distilled water of 500 ml. The result will be 0.1% alkaline solution. Then bring Roselle seeds oil 1 ml and mix with Isopropyl alcohol 10 ml and Phenolphthalein 0.25 ml, then drop sodium hydroxide 0.1% until it is purple-pink. Then bring (Number of drops) multiply (0.05) plus (3.50). It is the amount of alkalinity used per liter.

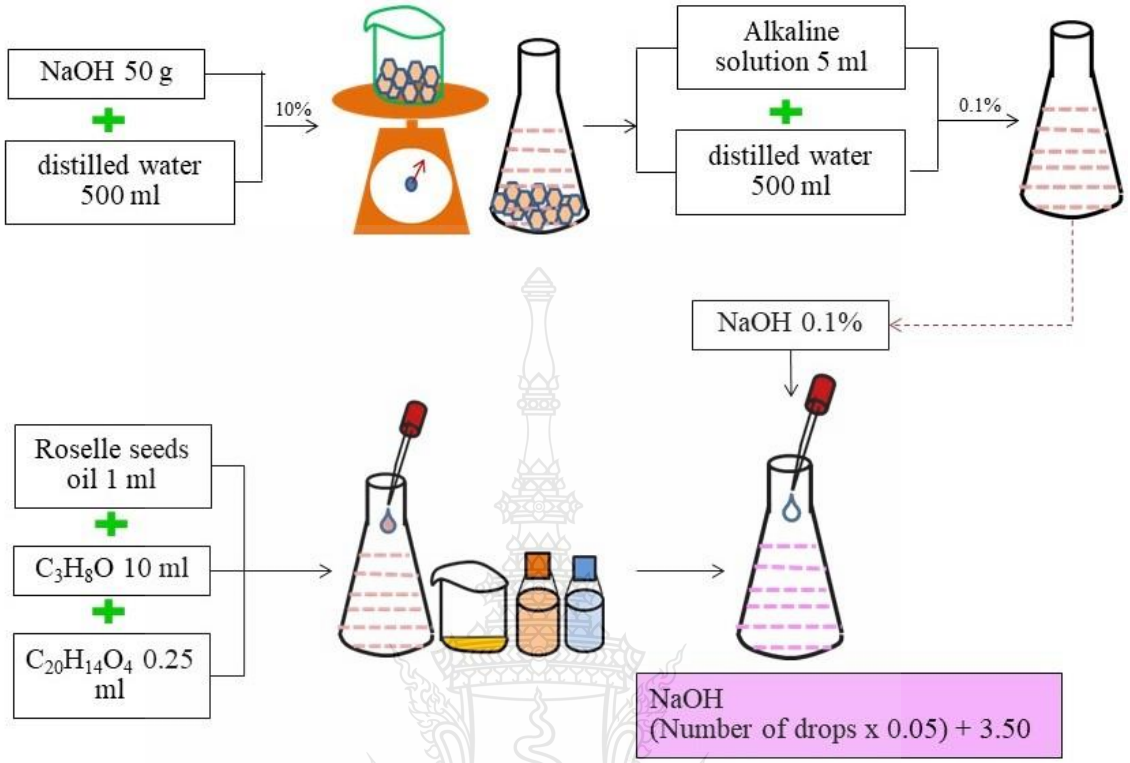

Figure 3.7 Titration method

3.8 Transesterification process

Figure 3.8 shows the method of producing biodiesel from Roselle seeds oil. Boil water repellant from Roselle seeds oil at a temperature of 110ºC until all of the steam is used. Later, mix a quantity of alkali titration with methanol 23% per the quantity of oil used in the manufacture from Roselle seeds. Blend into the reactor of Roselle seeds oil at temperature of 60ºC. Slowly, blend the mixed reaction for 1 hour. Leave it to separate the layers for 1 hour.

The compound is separated into 2 layers. The upper floor is biodiesel and lower is glycerin. Drain the Glycerin out. Next, insert concentrated sulfuric acid 96% volume 0. 19 ml and 982 ml of water. The acid catches up with a bass existing in biodiesel oil. This reduces soap in the washing water.

Therefore, open the air pump for 15 min leads to discarding water separation for 15min. Then drain out the remaining water but not biodiesel. Do the same again but no need to refill the acid.

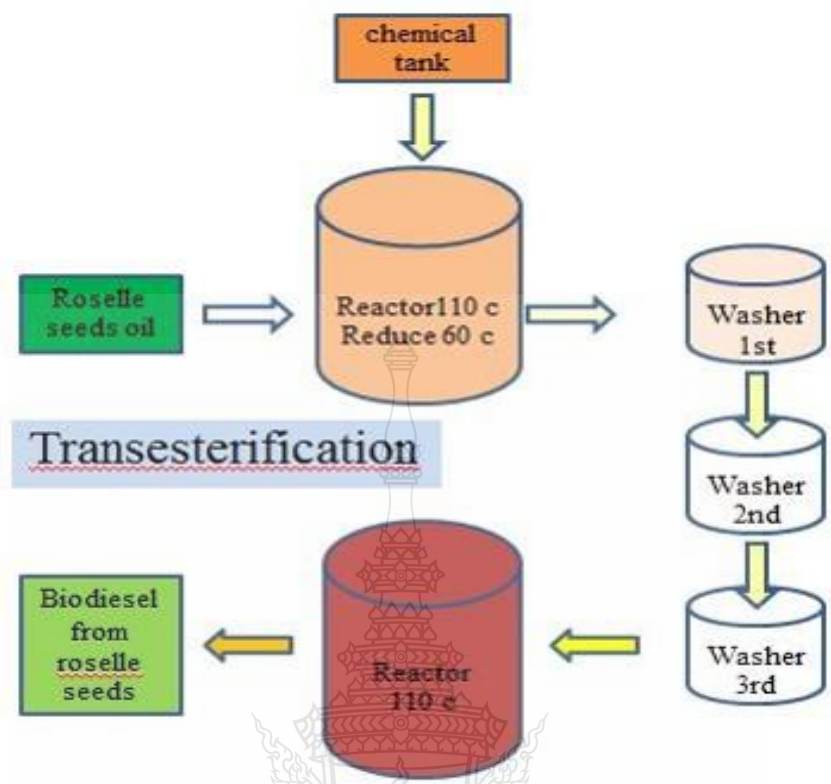

Figure 3.8 Transesterification process sample

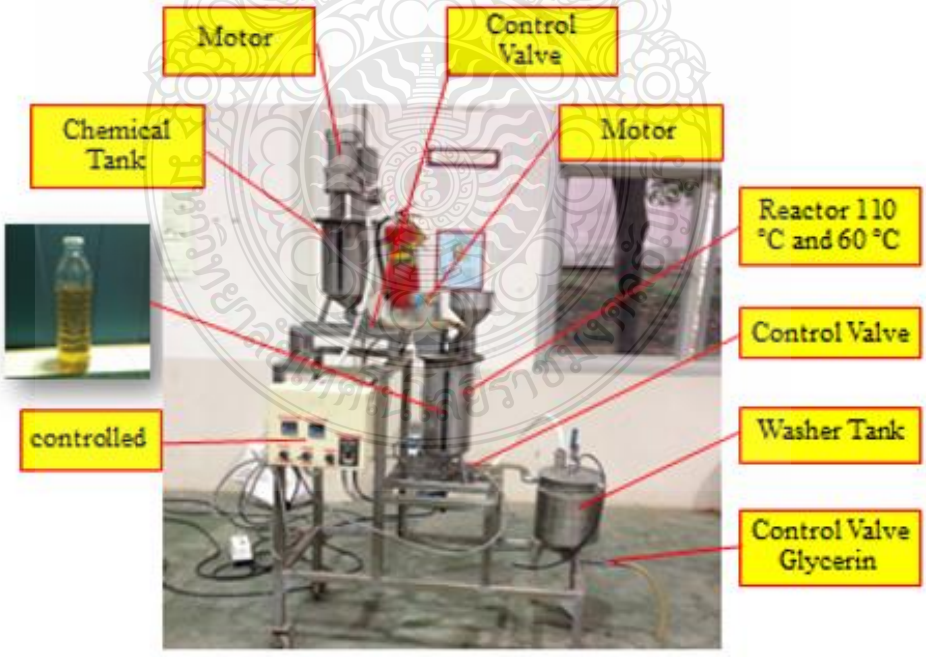

Figure 3.9 Sample of Biodiesel Machine.

CHAPTER4

RESULTS AND DISCUSSION

4.1 Bio-oil extraction from Roselle seeds

The moisture results from the six experiments for M8, M10, and M12 are shown in graph form below in Figure 4.1. The X-axis depicts the number of times the experiments were conducted and gave accurate information, while the Y-axis indicates the moisture present in the different Roselle seed sets. The average of water in the seed is 8% (M8), 10% (M10) and 12% (M12) with standard deviations (SD) of ± 0.15, ± 0.12 and ± 0.27 respectively. The seeds of the plant are 4mm wide, 5mm long, and 2.5mm deep. The crude oil extracted from the Roselle seeds is a yellow and viscous liquid before any distillation has taken place. The properties were checked to determine the appropriate production process for final use as a raw material in biodiesel production. The amount of oil received as a result of the oil extraction tests using the screw extractor on a constant 0.0468 m^3 of Roselle seed are shown below in Table 4.1. From the three moisture contents tested, the following yields of extracted Roselle seed oil and residue were found: M8 1300 ml, residue 4.36%; M10 1323 ml, residue 4.76%; M12 1274 ml, residue 5.93%. The overall comparison of the difference in the residue of the individual moistures shows an average of 1.57% for all the moisture residue of 5%, and the oil content extracted was 95%, due to the amount of crude oil obtained from the extraction with increasing amounts according to the specified humidity. Therefore, it is suitable for production as biodiesel.

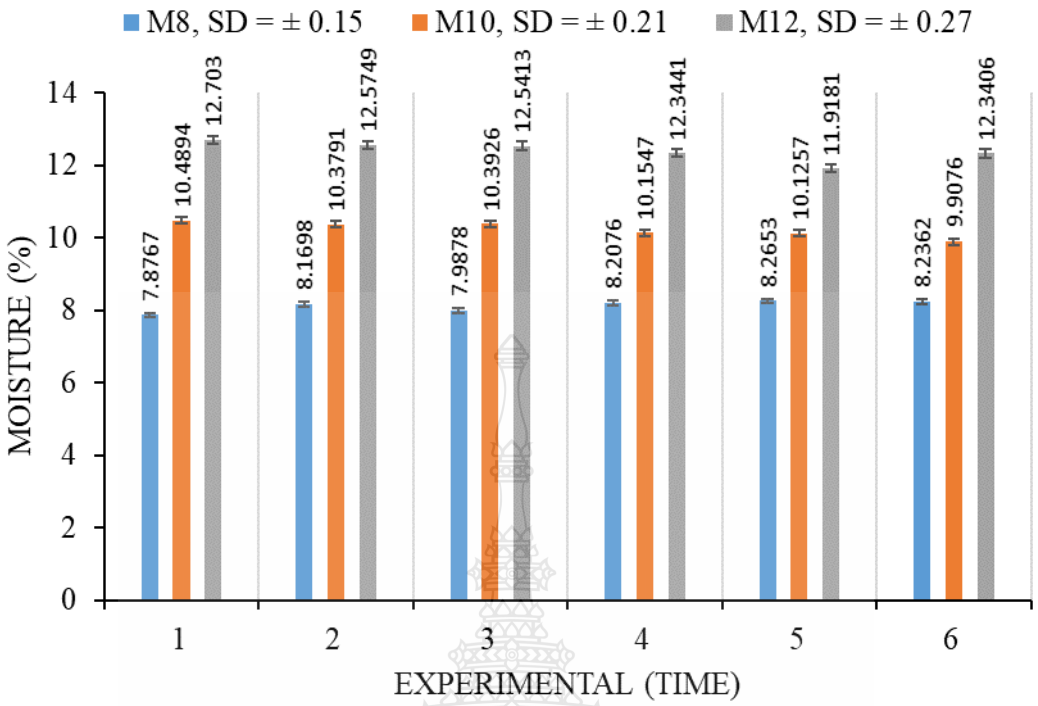

Figure 4.1 The Number of Experiments cf. Average Moisture.

Table 4.1 Comparison of Different Moisture Contents per Oil Content at Extraction

The moisture of Roselle Seeds (%)	The volume of Roselle Seed (g)	Roselle Seed Oil (ml)
M8	0.0468	1300
M10	0.0468	1323
M12	0.0468	1274

4.2 The analysis of Organic compound triglyceride

Table 4.2 below, shows the findings of the relationship of the surface moisture, volatile matter, ash, and fixed carbon produced when different heat conditions were applied to the three different moisture contents of Roselle seeds: M8, M10, and M12 respectively. The surface moisture is 4.00% wt., 3.96% wt., and 3.87% wt. The

decomposition of organic volatile matter is 72.47% wt., 73.01% wt., and 69.83% wt. The ash content is 7.14% wt., 6.92% wt., and 8.47% wt. The fixed carbon was calculated using the formula of Fixed Carbon = 100 – (Surface Moisture + Volatile Matter + Ash), which gave the following findings: 16.39% wt., 16.11% wt., and 17.83% wt., with a variance of ± 0.04% wt. Figures 4.2 to 4.4 all show the experimental DTA-TG. The green lines indicate how the mass given as a percent of the original sample changed during the test is relative to the temperature increase. The blue lines show the energy value at any point in time across the temperature range. Since volatile matter is caused by the decomposition of organic substances, triglycerides affect the formation of oils which are abundant in M8, M10, and M12 Roselle seeds of 72.47% wt., 73.01% wt., and 69.83% wt., respectively. Figure 4.5 shows the quantity of Surface Moisture, Volatile Matter, Fixed Carbon and Ash

Table 4.2 Results of Thermogravimetric Analysis

The moisture of Roselle Seeds (%)	% by Weight			
	Moisture Surface	Volatile Matter	Fixed Carbon	Ash
M8	4.00	72.47	16.39	7.14
M10	3.96	73.01	16.07	6.92
M12	3.87	69.83	17.82	8.47

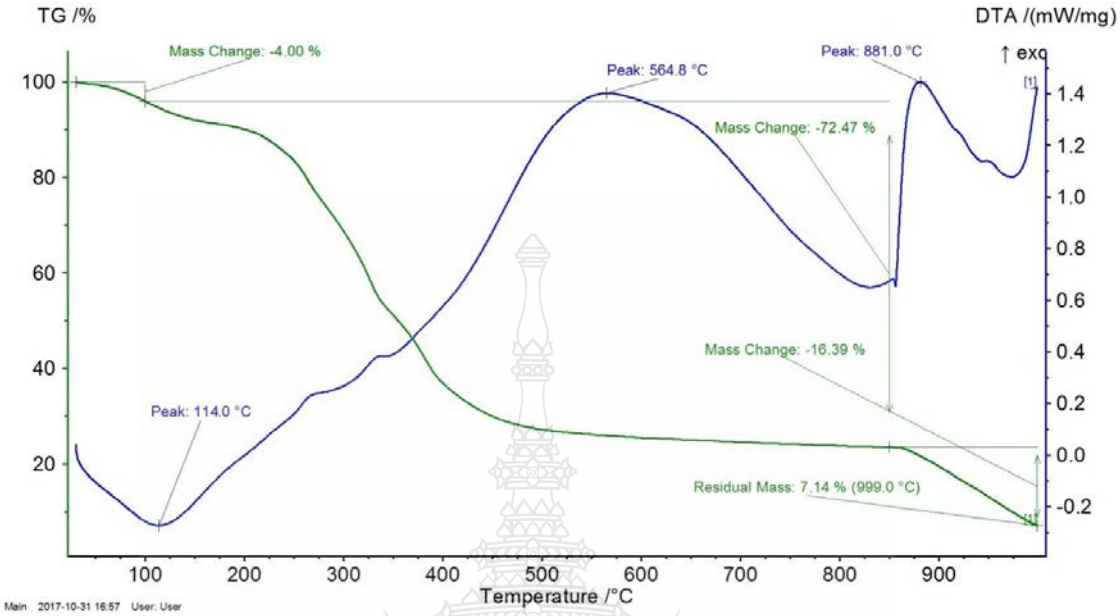

Figure 4.2 M8, DTA-TG Experiment

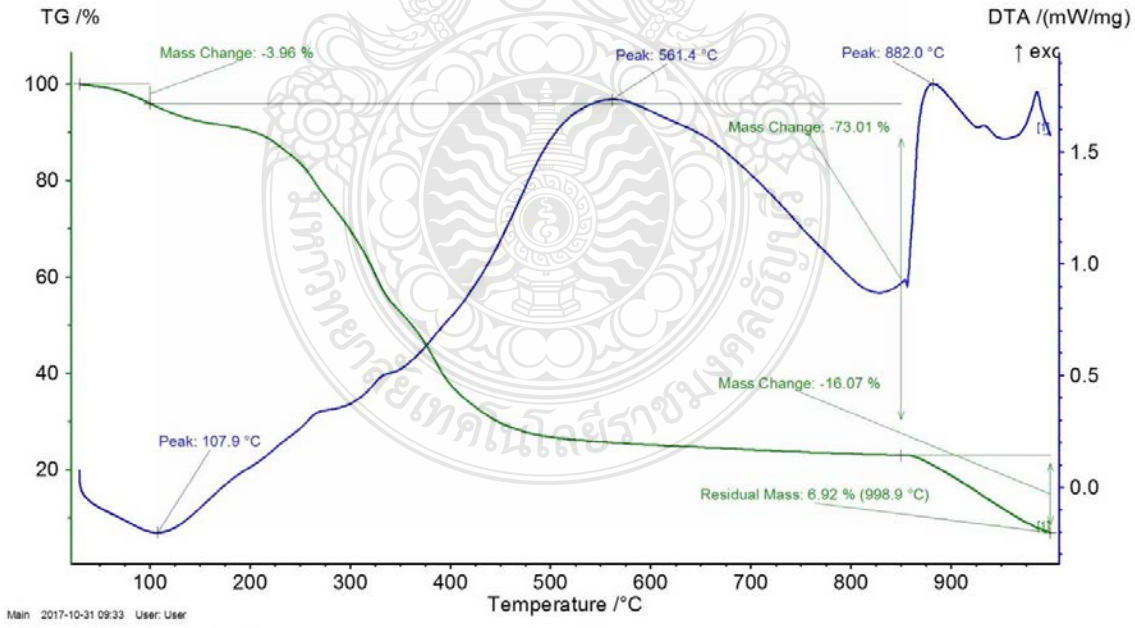

Figure 4.3 M10, DTA-TG Experiment

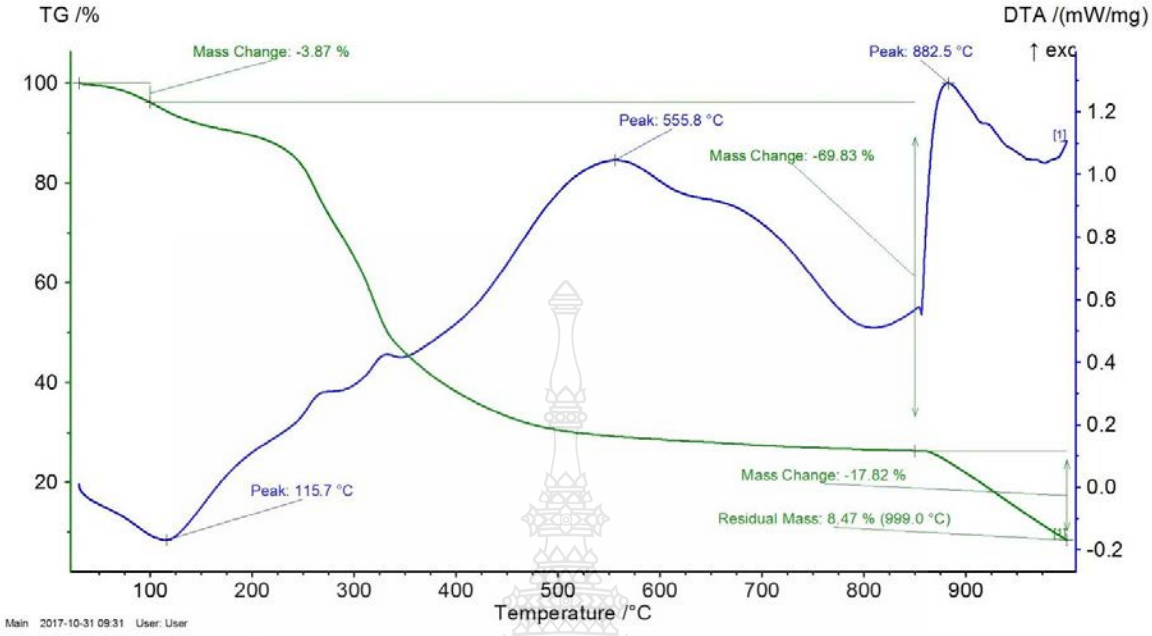

Figure 4.4 M12, DTA-TG Experiment

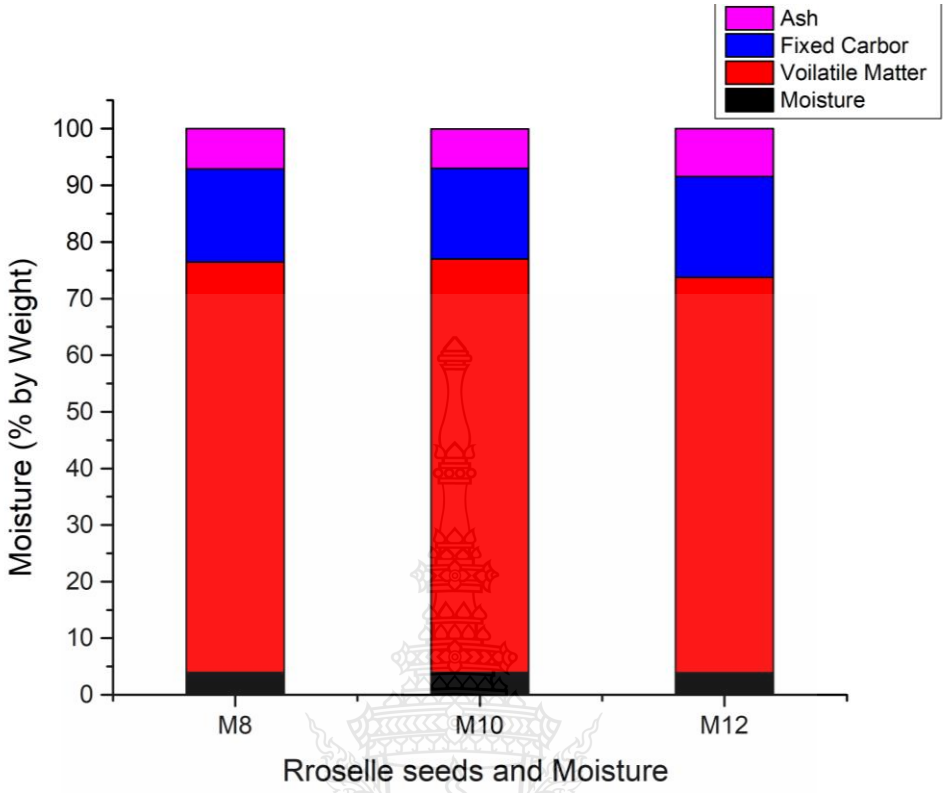

Figure 4.5 The show quantity Moisture Surface, Volatile Matter, Fixed Carbon and Ash

4.3 Bio-oil Characterization GC-FID M8, M10, M12

The standardized data of identification and dosage of fatty acids can be done by Supelco 37 FAME, compared with GC-FID data for Roselle seed oil FAME, that moisture M8, M10 and M12 divided into Palmitic acid and Stearic acid is Fatty acid groups (SAFA) of the saturated fats, Oleic acid Monounsaturated fats (MUFA), Linoleic acid polyunsaturated fats (PUFA) consisting of 18 carbon atoms. There are 2 pairs of bonds. The isomer type is an ester of triglycerides. Free fatty acids formed that does not combine with glycerol are triglycerides, g-Linolenic acid, a complex polyunsaturated fat (PUFA) which has 18 carbon atoms and 3 double bonds. It contains Omega 6. From tables 4.3, 4.4, and 4.5, maximum amount of fatty acids: Palmitic acid (22.46 %, M12) Stearic acid (5.04%) Oleic acid (47.73%) and g-Linolenic acid (2.71%). For Linoleic acid (33.33%, M8) FAME analysis based on the moisture content of Roselle seed oil has never been reported before.

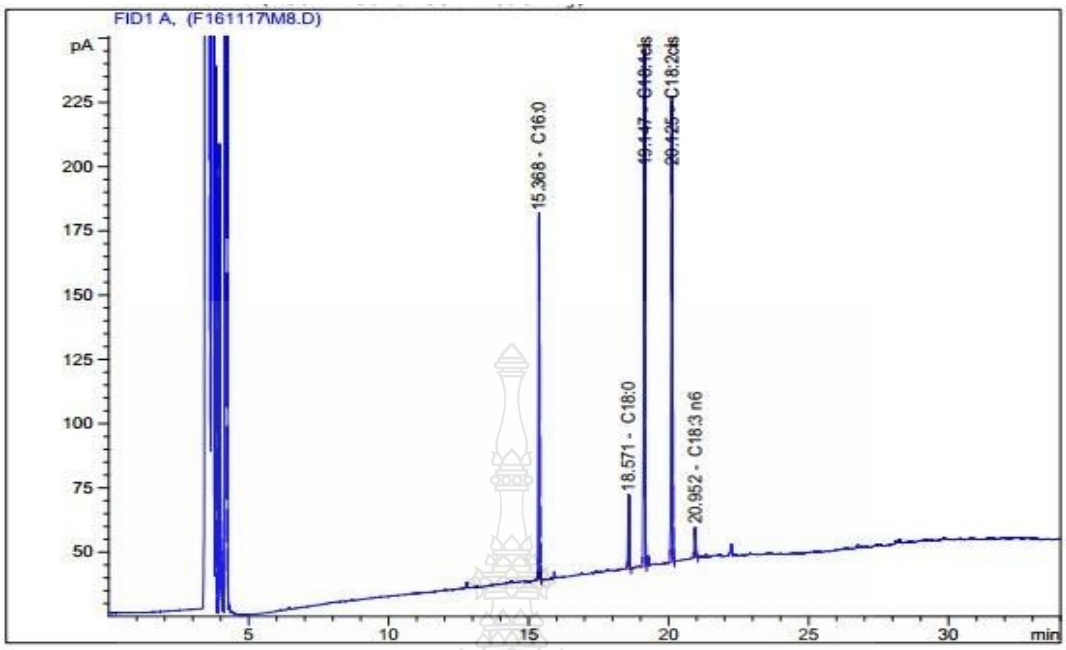

Figure 4.6 GC-FID Chromatogram of M8 with peak label (Retention Time and Name of Fatty Acid).

Table 4.3 Fatty acid methyl esters percentage in the area and fatty acid groups Roselle seeds, M8

Compounds	Fatty acids	Fatty acid groups	Retention time	Area	% Area
C16:0	Palmitic acid	SAFA	15.368	326.61	20.88
C18:0	Stearic acid	SAFA	18.571	75.50	4.83
C18:1 cis	Oleic acid	MUFA	19.147	606.91	38.80
C18:2 cis	Linoleic acid	PUFA	20.125	521.33	33.33
C18:3 n6	g-Linolenic acid	PUFA/ω6FA	20.952	33.83	2.16
		Total FAME		1564.18	100

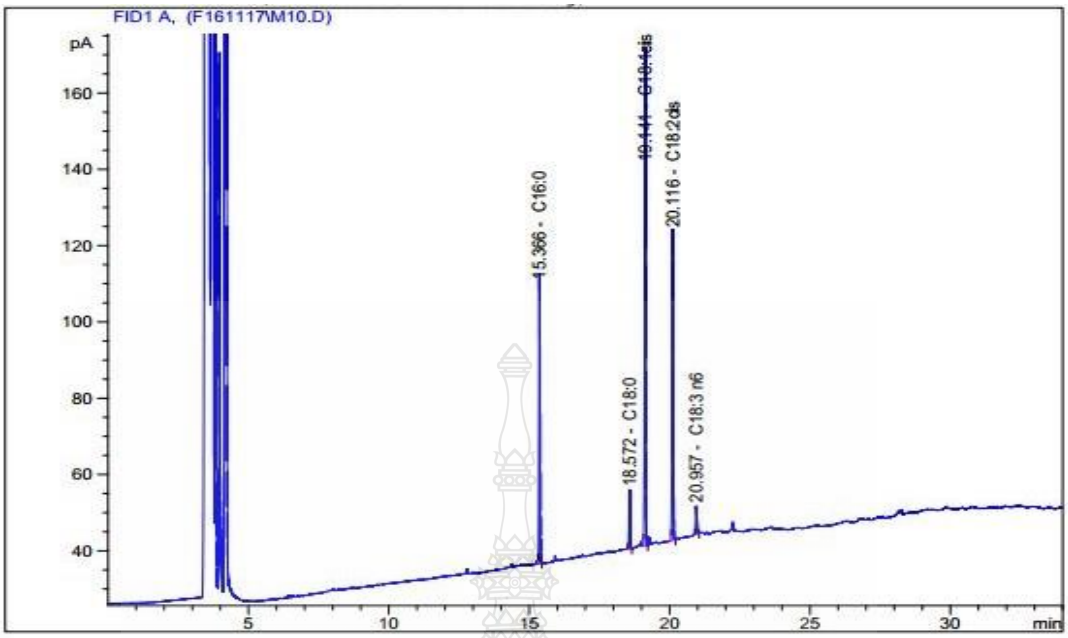

Figure 4.7 GC-FID Chromatogram of M10 with peak label (Retention Time and Name of Fatty Acid).

Table 4.4 Fatty acid methyl esters percentage in the area and fatty acid groups Roselle seeds, M10

Compounds	Fatty acids	Fatty acid groups	Retention time	Area	% Area
C16:0	Palmitic acid	SAFA	15.366	170.06	20.81
C18:0	Stearic acid	SAFA	18.572	40.75	4.99
C18:1 cis	Oleic acid	MUFA	19.141	358.92	43.92
C18:2 cis	Linoleic acid	PUFA	20.116	225.76	27.62
C18:3 n6	g-Linolenic acid	PUFA/ω6FA	20.957	21.73	2.66
		Total FAME		817.22	100

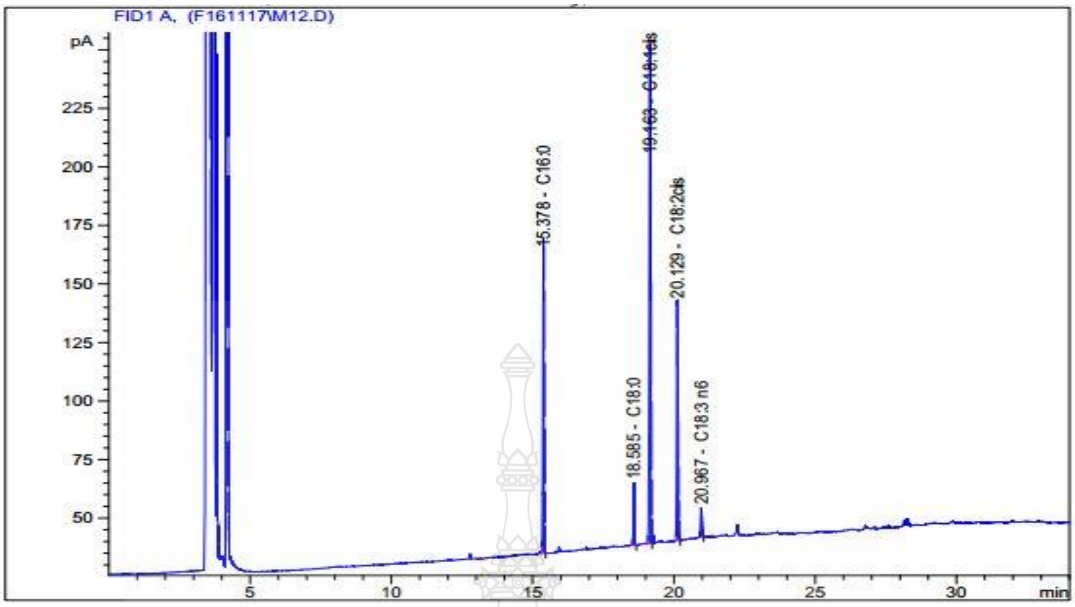

Figure 4.8 GC-FID Chromatogram of M12 with peak label (Retention Time and Name of Fatty Acid).

Table 4.5 Fatty acid methyl esters percentage in the area and fatty acid groups Roselle seeds, M12

Compounds	Fatty acids	Fatty acid groups	Retention time	Area	% Area
C16:0	Palmitic acid	SAFA	15.378	300.83	22.46
C18:0	Stearic acid	SAFA	18.585	72.40	5.04
C18:1 cis	Oleic acid	MUFA	19.163	639.29	47.73
C18:2 cis	Linoleic acid	PUFA	20.129	290.56	21.69
C18:3 n6	g-Linolenic acid	PUFA/ω6FA	20.967	36.31	2.71
		Total FAME		1339.39	100

4.4 The analysis of the properties of Bio-oil from Roselle seed

The properties of the extracted Roselle seed oil obtained in this study when compared with those of Jatropha oil obtained from [58], and also that of the Iodine value as can be seen in Table 4.6 below. Roselle seed oil can be changed using a methyl ester in the transesterification process through the reaction of methanol activated by a catalyst; in this case the sodium hydroxide alkaline. The transesterification process reduces the acidity, viscosity, Iodine, and density of the oil by altering the molecular structure to that of a larger size. The larger branch structure enables the resulting biofuel to have a higher Flashpoint value than that of the Jatropha oil. This result is of great benefit because it is more convenient for safer fuel storage. The pour point value of the Roselle seed oil is better than that of Jatropha oil. The freezing point of the produced oil is -3.5, which is lower than that of Jatropha oil, so it has a good pouring point, that has the potential to flow in a landscape with lower temperatures than the freezing point compared to Jatropha. This indicates that Roselle seed oil is much better than Jatropha oil and can be used to produce biodiesel.

Table 4.6 Comparison of Roselle and Jatropha Seed Oil

Property Tested	Roselle Seed Oil	Method	Jatropha Oil [58]	Method
Pour point (°C)	-3.5	ASTM D5950	4	ASTM D5950
Acidity (mg KOH/g)	32.45	ASTM D664	28.0	ASTM D664
Viscosity (c St)	41.05	ASTM D445	24.5	ASTM D445
Iodine (g I2/100 g)	40.05	EN 14111	92.0	EN 14111
Flash Point (°C)	278	ASTM D93	225	ASTM D93
Density (15°C, kg/m^3)	910	ASTM D4052	940	ASTM D4052

4.5 The calorific value of bio-oil

From table 4.7, The calorific value is a fundamental property of fuel. The amount of heat indicates energy value. Roselle seed oil provides a high calorific value of up to 39.38 MJ / Kg., compared with Jatropha oil = 38.65 MJ/Kg [58] and palm kernel oil = 37.50 MJ/Kg. Triglycerides contained in the volatile matter of roselle seeds has a good heating effect. As a result of GC-FID, it has a good effect on the calorific value since the Linoleic acid of substances found in roselle seed oil according to moisture M8, M10, and M12 has the following values 33.33%, 27.62%, and 21.69% respectively, which is abundant in roselle seed oil resulting in a very good thermal energy value.

Table 4.7 The calorific value of bio-oil, Roselle seed, Jatropha, and palm kernel.

Raw material	Calorific value (MJ/Kg)	Methods
Roselle seed oil	39.38	ASTM D 240-14
Jatropha oil [58]	38.65	ASTM D 240-14
Palm kernel oil	37.50	ASTM D 240-14

4.6 Characteristics of GC-FID Biodiesel from Roselle seed

From Table 4.8, when passing through the transesterification process, the area of Palmitic acidity = 88.83, Stearic acid = 21.42, Oleic acid = 184.71, Linoleic acid = 115.42 and g-Linolenic acid = 10.74, respectively, which is a good cause for biodiesel production to reduce the fatty acids of the oil to meet the ASTM and DIN standards.

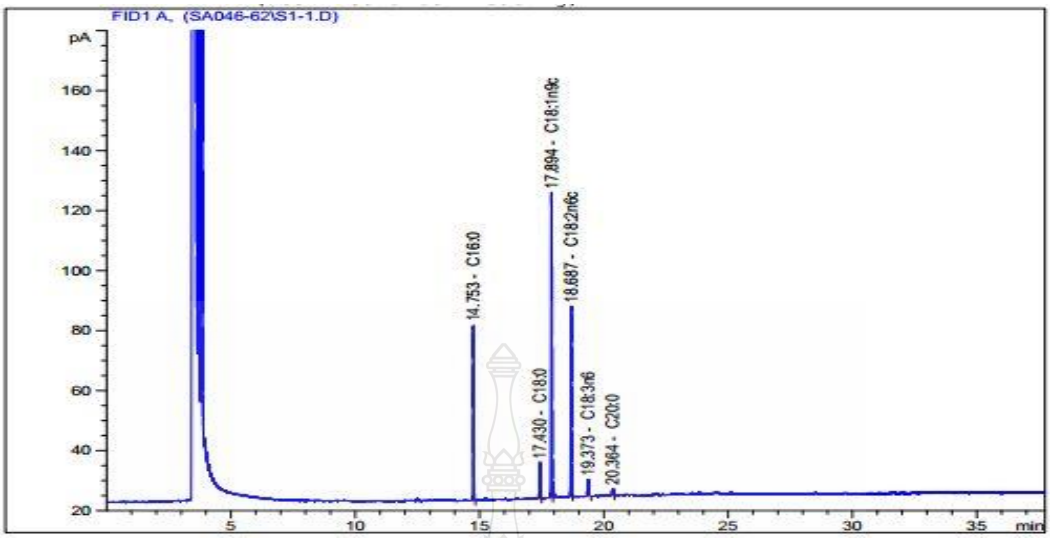

Figure 4.9 GC-FID Chromatogram Biodiesel of Roselle seed with peak label (Retention Time and Name of Fatty Acid).

Table 4.8 Biodiesel fatty acid of Roselle seeds, percentage in the area

Compounds	Fatty acids	Retention time	Area	% Area
C16:0	Palmitic acid	14.756	88.83	21.09
C18:0	Stearic acid	17.433	21.42	5.09
C18:1 n9c	Oleic acid	17.894	184.71	43.86
C18:2 n6c	Linoleic acid	18.689	115.42	27.41
C18:3 n6	g-Linolenic acid	19.379	10.74	2.55
	Total FAME		421.12	100

4.7 The analysis of the properties of Biodiesel from roselle seed

The Roselle seed oil when processed by Trasnsesterification to change the structure from Triglycerides is Mono alkyl Ester and Glycerine or Glycerol by reacting with methanol and sodium hydroxide is a catalyst from this study, when compared with biodiesel, jatropha, and diesel oil obtained from, see table 4.9 below. Biodiesel from Roselle seeds gave a Pour point = -1ºC, biodiesel from Jatropha = 2ºC showed that at low temperatures, biodiesel from Roselle seeds had better pouring. The acidity of biodiesel from Roselle seeds is in the standards of ASTM = <0.80 and DIN = <0.50, Viscosity value = 4.5 c St according to ASTM = 1.9-6.0, DIN = 3.5-5.0, Flash Point = 285ºC conforms to ASTM = >130, DIN = >120, which is higher than Jatropha biodiesel and diesel fuel is better and safe to use and transport. The density value = 860 kg / m^3 is a basic physical property, has properties very close to diesel, and more biodiesel from Jatropha shows that the efficiency of biodiesel from Roselle seeds is very good for water to replace diesel oil.

Table 4.9 Comparison Property of Roselle seed Biodiesel, Jatropha Biodiesel, and Diesel oil

Property Tested	Roselle seed Biodiesel	Jatropha Biodiesel[58]	Diesel[58]	Biodiesel Standards	
				ASTM D 6751-02	DIN EN 14214
Pour point (°C)	-1	2	-20	-	-
Acidity (mg KOH/g)	0.43	0.40	-	<0.80	<0.50
Viscosity (c St)	4.5	4.8	2.6	1.9-6.0	3.5-5.0
Flash Point (°C)	285	135	68	>130	>120
Density (15°C, kg/m^3)	860	880	850	-	860-900

4.8 The calorific value of biodiesel

From Table 4.10, the calorific value is the basic property of the fuel. The amount of heat refers to the energy value of biodiesel from Roselle seeds which has a calorific value of 39.27 MJ / Kg compared to Jatropha oil = 39.23 MJ / Kg and is similar to diesel fuel [58] is very hot.

Table 4.10 The calorific value of Roselle seed biodiesel, Jatropha biodiesel, and diesel.

Raw material	Gross heat of Combustion (MJ/Kg)	Methods
Roselle seed Biodiesel	39.27	ASTM D 240-14
Jatropha Biodiesel [58]	39.23	ASTM D 240-14
Diesel [58]	42	ASTM D 240-14

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

In this study, the effect of moisture content on the properties of extracted seed oil was determined. The result showed these details: The result on oil content, showed that lower moisture content (8%, 10%) gave the higher amount of oil content than higher moisture content (12%). The result on the volatile matter, fixed carbon, and ash content, showed that all of these moisture content (8%, 10%, 12%) gave the near value of the volatile matter, fix carbon and ash content value. The result on fatty acid composition from GC showed that different moisture content (8%, 10%, 12%) had more effect on oleic acid and linoleic acid than palmitic acid, stearic acid, and linolenic acid. The variation of the amount of oleic acid, linoleic acid is higher than palmitic acid, stearic acid, and linolenic acid. The combined extracted seed oil from different moisture content has a higher acid value of 4 mg KOH/g. So, it needs to neutralize this oil with the base to below 4 mg KOH/g of acid value. The obtained oil will further make biodiesel by using a transesterification reaction to produce biodiesel. The obtain roselle seed oil biodiesel had every property (pour point, acidity, viscosity, flash point, density) which met well with ASTM and DIN EN standard.

RECOMMENDATIONS

This research is to find biological raw materials or agricultural waste and analyze their basic qualifications and physical properties as well as the energy characteristics that will be obtained to use as a renewable fuel power to fossil fuels that are diminishing and insufficient in today's world, and substitute more suitable economic crops for food use, such as palm oil.

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APPENDICES

(List of Publications)

List of Publications

International Journal

1. **Homraruen K.,** and Chanpeng W., 21 (September 2021). Effect of Moisture Content on Roselle Seed Oils Properties and its Biodiesel Properties for Renewable Fuel. *Internation Energy Journal*

International Conferences

1. **Homraruen K.,** and Chanpeng W., 2018. Study of Property Basic and Moisture Content of The Roselle Seeds per Oil Extraction Volume and Moisture Relations Analyzer for Used as Raw Material Production of Biodiesel Renewable Fuel. *International Conference on Science and Technology of Emerging Materials, Pattaya, Thailand, 18-20 July.*

National Conferences

1. Chanpeng W., and **Homraruen K.,*** 2558. The oil extracted from the roselle seeds use as a feedstock for biodiesel production. *Thailand Renewable Energy for Community Conference CF007: 76-78.*

Biography Name - Surname	Kittapas Homraruen
Date of Birth	December 25, 1970
Address	44 Moo 2, Nongpling Sub-district, Laokhwan District, Kanchanaburi Province , Thailand 71210
Education	2014 Doctor of Engineering Program in Energy and Materials Engineering(International Program), Faculty of Engineering, Rajamangala University of Technology Thanyaburi (RMUTT) 2007 M.A. : Master of Science in Technical Education (Mechanical Technology) from King Mongkut's University of Technology North Bangkok, Thailand 1995 B.Ind.Tech: Bachclor of Industrial Tecnology in Mechanical Engineering from South-East ASIA University
Experience Work	2020 Lecturers Mechanical Engineering of Rajamangala University of Technology Suvarnabhumi. suphanburi, Thailand. 2011 Training for lecturers responsible energy of (building). Prathumthani, Thailand. 2005 Head of Blade manufacturing at (Thai Astomow). Chachoengsao. 2003 Factory Engineer for production company (Starsanitaryware), Saraburi,
Telephone Number	06-2514-6641
Email Address	kittapas57@hotmail.com Kittapas_@mail.rmutt.ac.th