MAKERERE UNIVERSITY

COLLEGE OF NATURAL SCIENCES

SCHOOL OF PHYSICAL SCIENCE

DEPARTMENT OF CHEMISTRY

EXTRACTION AND TRANS-ESTERIFICATION OF JATROPHA OIL INTO FATTY ACID METHYL ESTERS.

BY

NANDUJJA VALERIAN VANESSA

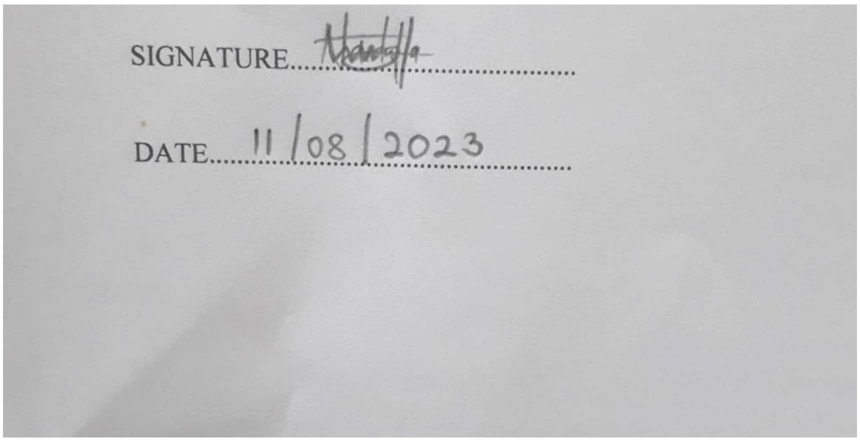
20/U/10152/PS

SUBMITTED TO THE DEPARTMENT OF CHEMISTRY, COLLEGE OF NATURAL SCIENCES IN PARTIAL FULLFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE BACHELOR OF SCIENCE IN INDUSTRIAL CHEMISTRY

August, 2023

DECLARATION

I, **NANDUJJA VALERIAN VANESSA**, here by declare that this is my final year project, with the help of my advisor **Dr. EGESA DAN.** This report is therefore my original work and the information embodied in it has not been submitted either in part or in full anywhere for any award in any manner. Where any other information has been used in this research, the authors have been acknowledged.



DEDICATION

I dedicate this report to all the people who have been of help to me to achieve the desired results of this project, my loving family, advisor and the entire staff of the Department of Chemistry. All these people have been affected in every way possible but have ensured that my final year project in which I have gained much knowledge about practical skills is a success, may God bless you abundantly.

ACKNOWLEDGEMENT

I extend my sincere gratitude to Mr. Kavuma from the advanced physical chemistry Laboratory who helped me carry out the laboratory experiments needed to achieve the objectives of my project. I like to also extend my gratitude to my loving mother and father for having been there throughout my academic journey and for providing all the financial and emotional support I needed to carry out my project.

Special thanks to my advisor Dr. Egesa Dan for all the support and input during the experiments which helped me compile this report. Most importantly, I thank the Almighty God for good health and protection during the estimated time period of my project.

APPROVAL

This report has been submitted with the approval of my university advisor;

DR. DAN EGESA.

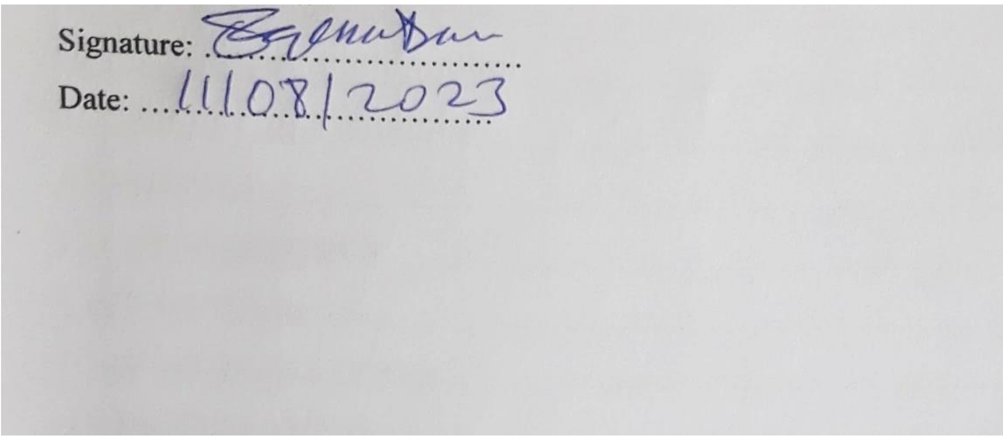


TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF EQUATIONS

ABBREVIATIONS

mL- millilitres

$H_2 SO_4$- Sulphuric Acid

g – grams

FAME – Fatty Acid Methyl Esters.

FFA – Free Fatty Acids

ABSTRACT

Biodiesel is a fuel that may be made from renewable resources, such as vegetable oil or animal fats, through the process of trans-esterification. It is non-polluting, accessible, sustainable, and reliable. On a bulk basis, its energy content is around 12% lower than that of petroleum-based diesel. When burned, biodiesel emits fewer pollutants than petroleum diesel. It reduces the severity of the greenhouse effect and does not increase the amount of carbon dioxide in the atmosphere.

The oil content is 25-30% in the seed making it potential for biodiesel production. The oil contains 21% saturated fatty acids and 79% unsaturated fatty acids. Oil has very high saponification value and being extensively used for making soap in some countries. Also oil is used as an illuminant in lamps as it burns without emitting smoke. It is also used as fuel in place of, or along with kerosene stoves. The oil was extracted using solvent extraction using n hexane as the solvent using 105g of the kernel powder. The mixture of the oil was separated using distillation and the oil (60ml) was residue. The oil extraction yield was 52.23% from this study. The biodiesel from jatropha oil is produced by trans-esterification from the extracted oil. This study was carried out to compare the biodiesel yield when trans-esterification is done in 2 steps with that of literature where only base catalyzed trans-esterification was used.

The fatty acid were reduced by reacting the 50ml of jatropha oil with a heated mixture of concentrated H_2 SO_4 and methanol (60^0 C), stirred for 1 hour and left to settle. The separated oil was then used for trans-esterification which was carried out on using prepared sodium methoxide, stirred for an hour and left to settle for 24 hours. The quantity of biodiesel was recorded to be 14.5ml.The yield was above average. Jatropha seed oil is a good feedstock potential for the production of biodiesel and at conditions of 1:6 methanol to oil ratio, 1 hour reaction time and 0.5g catalyst load the reaction yielded 14.9ml of the fuel. And 21ml of the product from the first step of trans-esterification were used to produce the final 14.9ml of FAME to give to give a yield of 70.952%. And the reduction of free fatty acids before trans-esterification increases the productivity.

CHAPTER 1 : INTRODUCTION

1.1 Background Of The Study

The interest in alternative fuel sources, notably biofuels, which are renewable and environmentally beneficial, has increased due to concerns about the quick depletion of petroleum oil and its effects on the environment. Concerns about using vegetable oils instead of petroleum or mineral oil as a source of material have grown over the past few decades. Because of its portability, availability, renewability, inherent lubricity, lower sulphur and aromatic levels, and inherent lubricity, biodiesel is preferred to diesel fuels (Ahmed, 2016).

Biodiesel is a fuel that may be made from renewable resources, such as vegetable oil or animal fats, through the process of trans-esterification. It is non-polluting, accessible, sustainable, and reliable. On a bulk basis, its energy content is around 12% lower than that of petroleum-based diesel. When burned, biodiesel (whether it is mixed or pure) emits fewer pollutants than petroleum diesel. It reduces the severity of the greenhouse effect and does not increase the amount of carbon dioxide in the atmosphere (Ahmed, 2016).

Jatropha curcas

Swedish botanist Carl Linnaeus initially described Jatropha curcas L. in 1753. It belongs to the numerous and diversified Euphorbiaceae family and is one of the many species in the genus Jatropha. Numerous Euphorbia species are renowned for producing phytotoxins and milky white sap (Brittaine, 2010).

Analysis of jatropha curcus seed shows the following chemical compositions.

Moisture: 6.20% Protein: 18.00% Fat: 38.00% Carbohydrates: 17.00% Fiber: 15.50% Ash: 5.30%

The oil content is 25-30% in the seed. The oil contains 21% saturated fatty acids and 79% unsaturated fatty acids. These are some of the chemical elements in the seed, cursin, which is poisonous and render the oil not appropriate for human consumption. Oil has very high saponification value and being extensively used for making soap in some countries. Also oil is used as an illuminant in lamps as it burns without emitting smoke. It is also used as fuel in place of, or along with kerosene stoves. Jatropha curcus oil cake is rich in Nitrogen, Phosphorous and Potassium and can be used as organic manure. By thermodynamic conversion process, pyrolysis, useful products can be obtained from the jatropha oil cake. The liquid, solid (char), and gaseous products can be obtained. The liquid can be used as fuel in furnace and boiler. It can be upgraded to higher grade fuel by transesterification (S.Antony Raja, 2011).

This biodiesel can be produced by using the seed oil from the Jatropha curcas plant, which is not edible.Jatropha curcas seeds can be processed to create high-quality biodiesel fuel since they contain 27–40% oil (Ahmed, 2016). Because it offers the desired physiochemical and performance qualities that are equivalent to diesel to enable continuous operation with little change in design, the non-edible vegetable oil of Jatropha curcas is a promising commercially viable alternative to diesel (Sayyar, 2009).

Lamps used to be lit by jatropha oil in the past. Rural people still utilize it now for local soap making and therapeutic purposes. The jatropha plant is used as a living fence in India and many African nations to keep out grazing animals. In Madagascar and Uganda, jatropha is grown to act as a physical support for vanilla plants (Brittaine, 2010).

Extraction of the oil.

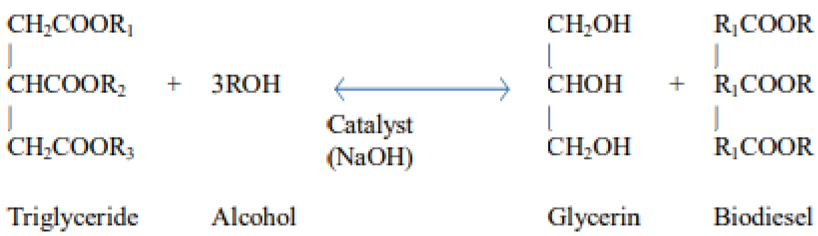
Soxhlet extraction has traditionally been used for a solid sample with limited solubility in a solvent in the presence of insoluble impurities. A porous thimble loaded with a solid sample is placed inside the main chamber of the Soxhlet extractor. By refluxing the solvent through the thimble using a condenser and a siphon side arm, the extraction cycle is typically repeated many times. Soxhlet extraction is a rugged, well-established technique and permits unattended extraction. However, it requires a long extraction time and the consumption of a large amount of solvent.

Soxhlet extraction has been used widely for extracting valuable bioactive compounds from various natural sources. In this extraction, a small amount of dry sample is placed in a thimble, which is placed in a distillation flask containing the solvent of particular interest. After reaching an overflow level, the solution of the thimble-holder is aspirated by a siphon, which unloads the solution back into the distillation flask. This solution carries the extracted solutes into the bulk liquid. The solute remains behind in the distillation flask, and the solvent passes back to the solid bed of samples. The process is repeated until complete extraction takes place (Saim et al., 1997).

Trans-esterification.

Trans-esterification is the process where vegetable oils and alcohol react with one another in the presence of a catalyst to replace an alcohol functional group from an ester with a different one. Oil is turned into biodiesel by this procedure. Methanol, ethanol, and propanol are all acceptable alcohols that are regularly used for this purpose, but methanol or ethanol use is more prevalent. The molar ratio of alcohol to oil, the catalyst concentration, the reaction time, the reaction temperature, the free fatty acid content, and the water content in oils or fats are all variables that might affect the trans-esterification process (Ahmed, 2016).

A triglyceride (fat/oil) reacts with an alcohol during the trans-esterification process to produce esters and glycerol. A triglyceride has three long chain fatty acids linked to the base molecule of glycerin. The type of fatty acids connected to the glycerin determines the properties of the fat. The properties of the biodiesel can be impacted by the fatty acid composition. Triglyceride and alcohol are combined during the esterification process in the presence of a catalyst, typically a potent alkaline substance like sodium hydroxide. Alcohol and fatty acids combine to create mono-alkyl ester, also known as biodiesel and crude glycerol.



Equation 1: chemical equation showing what happens during trans-esterification.

Trans-esterification process

The most common alcohol used in manufacturing is methanol or ethanol, which are base catalyzed by either potassium or sodium hydroxide (ethanol creates ethyl esters while methanol produces methyl esters). The methyl ester biodiesel reaction is below the chemical process. Alcohol must be provided in excess to the reversible interaction between the fat or oil and alcohol in order to move the reaction in the appropriate direction and guarantee complete conversion (Nitesh, 2020). There are three main methods for producing biodiesel from oils and fats:

1. Catalyst base transesterification of oil

2. Direct acid catalyst transesterification of the oil

3. Conversion of the oil to its fatty acid then biodiesel

Alcohol must be provided in excess to the reversible interaction between the fat or oil and alcohol in order to move the reaction in the appropriate direction and guarantee complete conversion. The separation of the ester and glycerol layers following the reaction time indicates a successful trans-esterification reaction (Nitesh, 2020).

1.2 Problem Statement

Uganda at present is heavily dependent on petroleum fuels for transportation and agricultural machinery. The fact that a few nations together produce the bulk of petroleum has led to high price fluctuation and uncertainties in supply for the consuming nations.Conventional fuels like coal also release gases such as sulfur dioxide, carbon dioxide, particulate matter, and other pollutants into the air and this pollution poses as a dangerous threat to the health of people. This has prompted the hunt for an alternative fuel that can be both environmentally friendly and sustainable. There is increasing environmental issues like global warming which leads to increase in temperatures and then flooding and other numerous issues, including growing fuel prices, resource depletion and energy sustainability (Dangoggo, 2018).

1.3 OBJECTIVES OF THE STUDY

1.3.1 General Objective

- Trans-esterification of Jatropha oil to FAME or biodiesel.

1.3.2 Specific Objectives

- Preparation of Jatropha seeds for extraction of oil.
- Extraction of oil from Jatropha seed crushed kernel.
- Trans-esterification of Jatropha oil to FAME or biodiesel.

1.4 SIGNIFICANCE

This project will help to check if Jatropha plant is a worthy candidate in being a source of oil that can be used in the production of biodiesel; a renewable and environmentally friendly fuel that can be substituted with the conventional fuels. This will improve the quality of life of Ugandans since it will reduce the consequences caused by pollution from using conventional fuels.

CHAPTER 2: LITERATURE REVIEW

The extraction of oil from Jatropha seeds has been attempted by different techniques such as solvent extraction by soxhlet extraction method (Dangoggo, 2018).

According to (Nitesh, 2020) biodiesels can be produced using different methods; Direct use of blending, Micro Emulsion process, Thermal Cracking (Pyrolysis) and Transesterification.

But Trans-esterification has been commonly used (Ahmed, 2016) (Alpandi, 2022) (Dangoggo, 2018) . Since Liquid base-catalyzed transesterification has certain limitations according to the amount of fatty acids (FFA's), soap formation and catalyst separation.So in order to overcome these short comings, liquid acid catalysts have been proposed for the transesterification reaction. Sulfuric acid (and hydrochloric acid have been most commonly used homogeneous acid catalysts. It has been reported that acid catalysts can be applied where the free fatty acid content of the raw material is high. Acid catalysts do not get affected by the presence of free fatty acids like alkali catalysts. But unfortunately, acid-catalyzed transesterification reactions usually have slower reaction rates with relatively lower conversion ratios,need a catalyst separation step, and have environmental as well as corrosion related problems according to (Deshpande, 2016).

The base catalyzed trans-esterification method was applied to produce biodiesel from Jatropha seed oil (Ahmed, 2016) at varied conditions; reaction time, catalyst concentration, alcohol reaction. The results obtained showed that a methanol to oil molar ratio of 4 with KOH concentration of 2.5% w/w and 75 min reaction time gave the optimum yield of biodiesel.

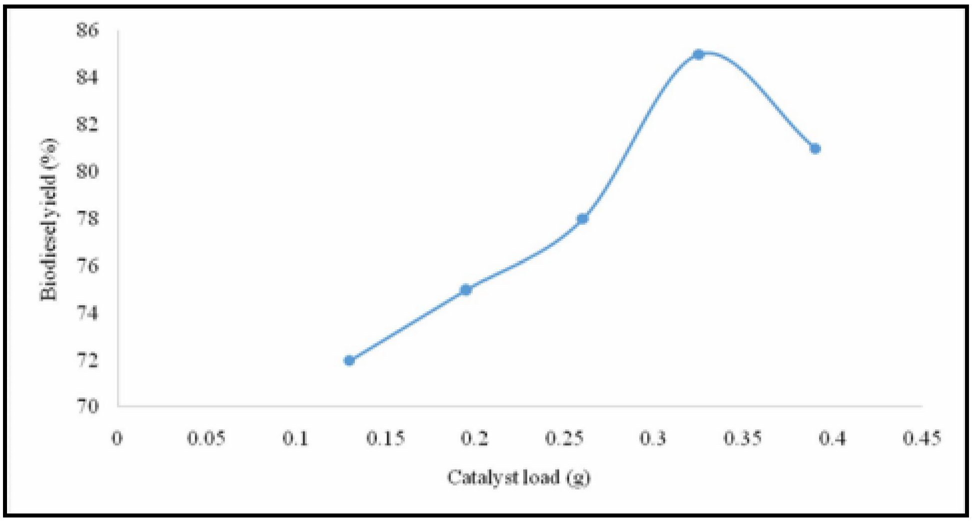


Figure 2.1: Biodiesel yield versus catalyst load (T = 65 oC, MOR = 4, t = 75 min, Voil = 15 mL) (Ahmed, 2016)

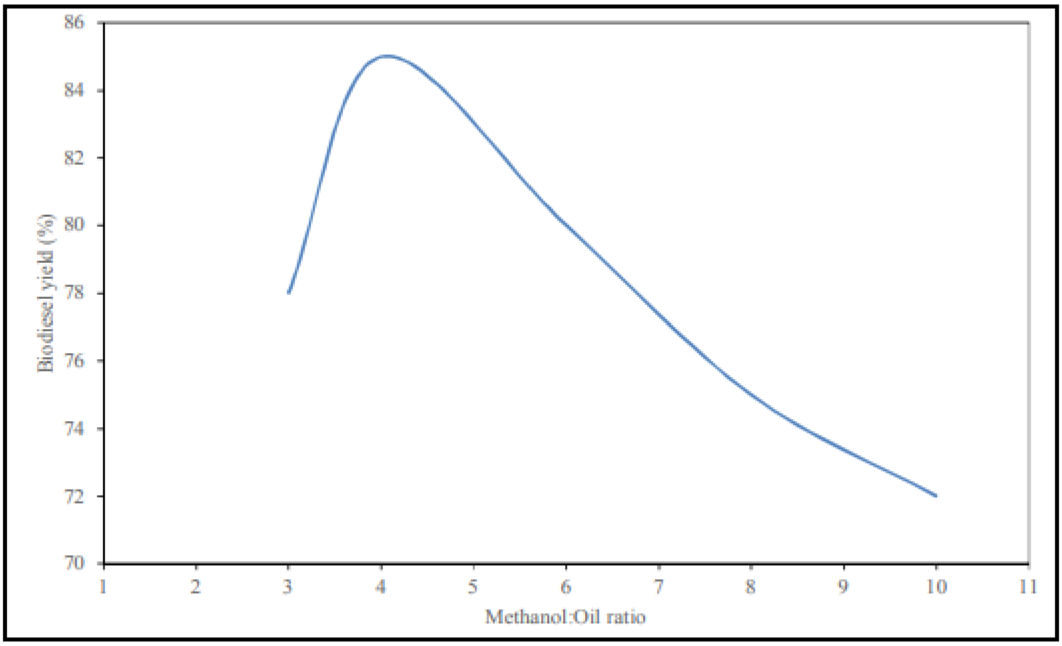


Figure 2.2: Graph of biodiesel yield against molar ratio (Catalystload = 0.325 g, T = 65 oC, t = 75 min, Voil = 15 mL) (Ahmed, 2016)

CHAPTER 3: MATERIALS AND METHODS

3.1 Reagents, apparatus, and materials

Thermometer, retort stand, pipette, measuring cylinder, separating funnel, magnetic stirrer, oven, water bath, hydrometer, conical flask, digital weighing balance, stop watch, hot plate, distilled water, methanol, and jatropha oil. methanol, Concentrated HCl, Concentrated sulfuric acid, Hexane, Magnetic stirrer.

3.2 Methods

3.2.1 **Preparation of the Jatropha seeds**

The Jatropha seeds were harvested from the plants. And then dried under the sun 4 hours for 3 days. The dried seeds were cleaned and removed from the mesocarps. The shells and husks of the seeds were cracked and carefully removed to get the inner kernels. The inner kernel was crushed using a blender.

Figure 3.1: sun drying the seeds.

Figure 3.2: Dry seeds of jatropha

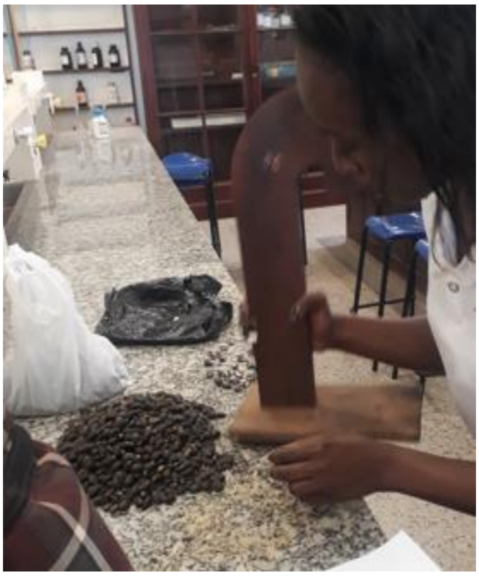


Figure 3.3: Separation of the kernel and mesocarp of the seeds.

Figure 3.4: The inner kernel ready to be crushed.

Figure 3.5: Blended kernel ready for oil extraction.

3.2.2 **Extraction of Oil.**

Oil was extraction using the soxhlet apparatus. A porous thimble loaded with a solid sample was placed inside the main chamber of the Soxhlet extractor. By refluxing the solvent through the thimble using a condenser and a siphon side arm, the extraction cycle was typically repeated many times at 70^0C.The mixture of solvent and oil was separated using distillation at 70^0C. The oil was the residue and it was left to cool and settle. The oil was kept in a sample bottle.

Figure 3.6: Extraction of oil using the soxhlet apparatus.

Figure 3.7: sample bottle with the collected oil from distillation residue showing a closer and distant view.

3.2.3 **Trans-esterification.**

Step 1

50ml of crude Jatropha oil was poured into a conical flask and heated to a temperature of 60°C. A mixture of 50ml concentrated H_2SO_4 (1% w/w) with 65ml methanol (30% v/v) was heated separately at (50°C) and then added to the heated oil in the flask. The mixture is stirred for 1 hour and allowed to settle for 2 hours. Using a separating funnel, the product was collected.

Step 2

21 ml of the product obtained from step 1 was measured and poured into 250 mL conical flask and heated to a temperature of 50°C.The sodium hydroxide pellet was placed in the weighing balance to get exactly 0.5 g. A solution of sodium methoxide was prepared in a

250 mL beaker using 0.5 g of sodium hydroxide pellet and 63 mL of methanol. The solution was properly stirred until sodium hydroxide pellet was completely dissolved. The sodium methoxide solution was placed in the cornical flask and heated to 60°C.The sodium methoxide solution was then poured into the warm Jatropha oil and stirred vigorously for 60 minutes using a magnetic stirrer. The mixture was then allowed to settle for 24 hours in a separating funnel. The biodiesel is then poured into a separate beaker, while the lower layer (which comprises of glycerol and soap) is collected from the bottom of the separating funnel. The sample was dried by placing it on a hot plate and excess water in the biodiesel was removed. The quantity of biodiesel collected was measured and recorded.

Figure 3.8: Upper layer of the product after tapping off the lower layer.

Conversion ratios of oil to biodiesel was determined on the basis of the ratio of obtained methyl ester amount to used oil amount (w/w) and decribed as biodiesel productivity (%).

CHAPTER 4: RESULTS AND DISCUSSION

4.1 RESULTS

Table 4.1: Results of the different methods

Preparation of seeds		Extraction of oil		Trans-esterification	
Obtained amount(g)	Used amount(g)	Obtained amount(ml)	Used amount(ml)	Amount of oil used(ml)	Amount of biodiesel obtained
450	105	60	50	21	14.9

The amount of oil extracted from 105g of the seed gave 60ml of oil. From these experiments 14.5ml of biodiesel was obtained from 21ml of the first product. The FFA's were reduced by acid catalyzed esterification. Acid catalyzed esterification gave a yield of 84%.

<u>Calculation of the oil and biodiesel productivity</u>

Yield = % Weight of sample /Weight of oil %Yield x (100)

Equation 2: yield of conversion.

Weight of sample used = 105g

Weight of oil obtained = 60ml

0.92kg/m^3(S.Antony Raja, 2011)

Mass = density x volume

=55g

From equation4.1

Yield = 55g/105g x 100%

52.38%

Yield of Trans-esterification

Step 1

Amount of oil used – 50ml

Amount of product – 42ml

Yield = (from equation 4.1)

42ml/50ml x 100%

=84%

Step 2

Amount of product used – 21ml

Amount of biodiesel obtained – 14.9ml

Yield = (from equation 4.1)

14.9ml/21ml x 100%

=**70.95%**

4.2 DISCUSSION

Preparation of the seeds was a success. Most of the seeds were not damaged. The seeds dried well.

The oil extraction had a yield of 52.38%. This is above average but still low. It should have lowered by the retention time for extraction (5 hours), solvent (n-hexane) and temperature conditions (70^0 C). But comparing with literature 47.5% (W.Ntalikwa, 2021), this oil extraction was a success.

Acid catalysed trans-esterification gave a yield of 84% and the second step which was base catalysed gave a yield of 70.952%.

From (Ahmed, 2016), base catalyzed trans-esterification was used to produce biodiesel from jathropha oil. The results obtained showed that a methanol to oil molar ratio of 4 with KOH concentration of 2.5% w/w and 75 min reaction time gave the optimum yield of biodiesel. Biodiesel yield was found to reduce with increasing KOH concentration and higher methanol to oil ratio.

This figure 2.1 shows that a higher catalyst load reduces biodiesel yield and so one of the reasons as to why this experiment gave a low yield could have resulted from the quantity of catalyst that was used which was 0.5g. Increased catalyst load gave way to production of other unwanted products from the reaction.

This figure 2.2 shows that a higher methanol ratio reduces biodiesel yield and so one of the reasons as why this experiment gave a low yield could have resulted from the quantity of alcohol that was used which was a molar ratio to oil of 6:1.

The results from trans-esterification qualified the method suitable to produce biodiesel.

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

1.5 CONCLUSION

In conclusion just like other researchers, Jatropha seeds oil is a good source of non-edible oil which is a potential feedstock for biodiesel. These results also confirm that n-hexane is a suitable solvent for the extraction of Jatropha oil under the conditions; extraction time of 5 hours, and extraction temperature of 70°C, where the oil yield was 52.23%. Basing on the results obtained, solvent extraction is indeed a good method for oil extraction from various plants for oils.

Jatropha seed oil is a good feedstock potential for the production of biodiesel and at conditions of 1:6 methanol to oil ratio, 1 hour reaction time and 0.5g catalyst load the reaction yielded 14.9ml of the fuel. 21ml of the product from the first step of trans-esterification were used to produce the final 14.9ml of FAME to give to give a yield of 70.952%. And the reduction of free fatty acids before trans-esterification increases the productivity.

The results show that trans-esterification is a suitable method for producing a biofuel from oil.

.

1.6 RECOMMENDATIONS

- I recommend that the initial stage which involves reduction of free fatty acids should be researched more.
- Another research can be done to investigate the characteristics of the residue meal after oil has been extracted because it might be potential fertilizer due to the presence of proteins.

CHAPTER 6: REFERENCES

1. Sepidar Sayyar, Zurina Zainal Abidin, Robiah Yunus and Azhari Muhammad (2009). Extraction of Oil from Jatropha Seeds-Optimization and Kinetics. *American Journal of Applied Sciences 6 (7)*, 1390-1395
2. Richard Brittaine, NeBambi Lutaladio (2010) . Jatropha: A Smallholder Bioenergy Crop. Integrated Crop Management .Vol. 8.
3. Mr. Baraiya Nitesh, Mr. Viral Chavan, Mrs. Vrushali Surve (2020). Production Of Biodiesel From Waste Cooking Oil. *International Journal of Creative Research Thoughts* , 2320-2882
4. Lake Belete Adamu, and Kamil Dino Adem(2020). Quality and Performance Evaluation ofJatropha Oil Blended with Kerosene for Cooking Stoves in Ethiopia, *Journal of Renewable Energy*, 1-9.
5. Amni Haslinda Alpandi, Hazlina Husin , Akhmal Sidek and Muslim Abdurrahman(). Characterization of Malaysian Jatropha Seed Oil and Discovering the Process of Powdered Jatropha Leaves. *Processes*, 1-14.
6. J. García-Dávila , E. Ocaranza-Sanchez, C. Sánchez and A. L. Martínez-Ayala(2018), Catalytic Activity of a Bifunctional Catalyst for Hydrotreatment of Jatropha curcas L. Seed Oil. *Journal of spectroscopy*, 1-7.
7. Ayten Sagiroglu, Şebnem Selen Isbilir, Hakki Mevlut, Ozcan Hatice Paluzar, Neslihan M. Toprakkiran(2011). Comparison Of Biodiesel Productivities Of Different Vegetable Oils By Acidic Catalysis. *Chemical Industry & Chemical Engineering Quarterly*, 53-58.
8. Justin W. Ntalikwa(2021). Solvent Extraction of Jatropha Oil for Biodiesel Production: Effects of Solvent-to-Solid Ratio, Particle Size, Type of Solvent, Extraction Time, and Temperature on Oil Yield. *Journal of renewable energy*, 1-8.
9. Hanumanth Mulimani, Dr. O D Hebbal, M. C. Navindgi (2012).Extraction Of Biodiesel From Vegetable Oils And Their Comparisons. *International Journal Of Advanced Scientific Research And Technology*, 242-250.
10. S.Antony Raja, D.S.Robinson smart, and C.Lindon Robert Lee ().Biodiesel production from jatropha oil and its characterization. *Research Journal of Chemical Sciences*, 81-87.

11. Patel Shivani, Patel Khushbu , Nilkanth Faldu , Vasudev Thakkar and R. B. Shubramanian (2011). Extraction and analysis of Jatropha curcas L. seed oil. *African Journal of Biotechnology 10(79),* 1810-1813.
12. Dangoggo SM, Dhikrah I, Sani NA, Baki AS, Bagudo BU and Jibrin MS (2018).Preparation and Characterization of Biodiesel Produced from Jatropha Seed Oil Using Sulphated Zirconia as Catalyst. *Ind Chem an open access journal*, 1-5.
13. Shriyash Rajendra Deshpande (2016). Production of Biodiesel from Soybean Oil Using Supercritical Carbondioxide, 1-82.
14. Konica Sarker (2016).Review and Comparison of Various Properties of Jatropha oil Biodiesel. *International Journal of Engineering and Technology*, 1965-1971.
15. Fahad Ahmed , Saidat Olanipekun Giwa, Maryam Ibrahim and Abdulwahab Giwa (2016). Production of Biodiesel from Jatropha Curcas Seed Oil using Base Catalysed Transesterification*. International Journal of ChemTech Research* 9(6), 322-332

APPENDICES.

1. **FAME**

These are esters of fatty acids. And are Fatty acids methyl esters.

2. **Trans-esterification.**

   This is the process of exchanging the organic functional group "R" of an ester with an organic group of "R" of an alcohol.

3. **Triglyceride.**

   Ester composed of free fatty acid units linked to glycerol.