### بسم الله الرحمن الرحيم

## **Sudan University of Sciences and Technology**

## **College of Graduate Studies**



## Extraction, and Physicochemical Characterization of the Oil Biodiesel and blends from seeds of *Ricinus Communis* (Castor Beans)

إستخلاص وتوصيف الخصائص الفيزيائية والكيميائية لزيت الوقود الحيوي ومخاليطه من حبوب الخروع

A Thesis submitted in fulfillment for the requirements of the degree of Doctor of Philosophy in Chemistry

By

**OMALHASSAN ALL SATTI SALEH (M.Sc., Chemistry)** 

Supervisor: Prof Mohammed El Mubark Osman

Co. Supervisor: Dr. Elfatih Ahmed Hassan

December 2020

## الإستهلال

## بسم الله الرحمن الرحيم

( فَبَحَا لَ بِأَوْعِيَتِهِمْ قَبْلَ وِعَاء أَخِيهِ ثُمَّ السَّهَ فَرَبَهَا مِن وِعَاء أَخِيهِ كَذَاكَ كِدْنَا السَّهَ فَرَبَهَا مِن وِعَاء أَخِيهِ كَذَاكَ كِدْنَا الْمَلِكِ لِيُوسُهُ مَا كَانَ لِيَأْدُذَ أَذَاهُ فِي حِينِ الْمَلِكِ لِيُوسُهُ مَا كَانَ لِيَأْدُذَ أَذَاهُ فِي حِينِ الْمَلِكِ إِلاَّ أَن يَشَاء اللَّهُ نَرْفَعُ حَرَبَاتٍ مِّن نَشَاء إِلاَّ أَن يَشَاء اللَّهُ نَرْفَعُ حَرَبَاتٍ مِّن نَشَاء وَفَوْقَ كُلِّ خِي عِلْمِ عَلِيمٌ ).

صدق الله العظيم

(بوسهم: 76)

## Dedication

To my parents, husband, daughters and brother.

To the soul of Asma Satti.

#### **Acknowledgements**

Praise to Allah Almighty who gave me health and patience to complete this work.

I would like to express my sincere gratitude to my supervisor Prof. Mohammed EL Mubark Osman for the continuous support, patience, motivation, and immense knowledge. His guidance helped me in stages of this of research.

My sincere thanks also go to my co-supervisor Dr. Elfatih Ahmed Hassan.

I would also like to thank Tarig Eltayeb Adam, Khartoum Refinery Company and Reem Hassan IshagAlgaddal, Sudan University of Science and Technology, Central Research Laboratory for their technical support.

#### **Abstract**

This study aims to obtain a sustainable source of energy from natural and environmentally friendly resources.

*Ricinus communis* seeds collected from Dongola city, northern state – Sudan were used as raw material for preparing biofuels and blends.

The oil was extracted from *Ricinus Communis* seeds using different techniques: chemical solvent extraction by soxhlet, cold chemical extraction with solvent and mechanical pressing. Percent oil yields were 49.0%, 35.5% and 31.2%, respectively. Gc/mass analysis showed the oil contained 82.14% Saturated fatty acids, 17.85% unsaturated fatty acids. The degree of saponification was 165.5 mgKOH/g, and it was found that the density value and the gravity factor are equal.

Biofuels were prepared by two methods, acid catalyzed esterification reaction and alkali-stimulated esterification using sodium hydroxide. Gas chromatography technique/mass spectrometer demonstrated that *Ricinus Communis* oil is converted biofuel.

The physical and chemical properties of the Biodiesel were verified according to the American Standard Test No. D 6751 and it was found that biofuel had met all of the standard requirements of the standard.

The mixture of *Ricin's Communis* fuel with fossil diesel No. D20 has been tested according to American Specifications No. D 7467 and has successfully passed the requirements of the standard. Also, two types of biofuel blends were prepared Biodiesel (20/80), fossil diesel and Biodiesel fossil diesel and ethanol (20/60/20) were prepared and compared with the ASTM No. D 7467. The three types of mixture met the standard requirements except for viscosity testing, where the viscosity level of biofuel fuel was 17.37 C<sup>0</sup> higher than the ASTM.

The acid number ratio was slightly higher than the standard and it was 0.6 mgKoH/g.

#### المستخلص

تهدف هذه الدراسة إلى الحصول على مصدر مستدام للطاقة من الموارد الطبيعية والصديقة للبيئة.

تم جمع بذور نبات الخروع من مدينة دنقلا - الولاية الشمالية - السودان كمادة خام لتحضير الوقود الحيوي ومزيجه.

تم استخلاص الزيت من بذور الخروع باستخدام تقنيات مختلفة وهي استخلاص المذيبات الكيميائية عن طريق السوكسليت و استخلاص المواد الكيميائية مع المذيبات و الضغط الميكانيكي. و بلغت نسبة المستخلاصات 49.0% و 35.5% و 31.2% على التوالي. أظهر تحليل Gc/mass أن الزيت يحتوي على 82.14% من الأحماض الدهنية المشبعة و 17.85% من الأحماض الدهنية غير المشبعة. كانت درجة التصبن 165.5mg/KOH ، وتبين أن قيمة الكثافة وعامل الجاذبية متساويان.

تم تحضير الوقود الحيوي بطريقتين من تفاعل الأسترة المحفز بالحامض والأسترة المحفزة بالقلويات باستخدام هيدروكسيد الصوديوم. أظهر مطياف الكتلة بتقنية الكروماتوغرافيا الغازية أن زيت الخروع تم تحويله إلى وقود حيوي.

تم التحقق من الخصائص الفيزيائية والكيميائية للوقود الحيوي وفقًا للمعايير القياسية الأمريكية رقم D6751 و وجد أن الوقود الحيوي قد استوفى جميع المتطلبات القياسية للمعايير. تم اختبار خليط الوقود الحيوي لزيت الخروع مع الديزل الأحفوري رقم D20 وفقًا للمواصفات

الأمريكية رقم D7467 ونجح في اجتياز متطلبات المعيار.

كما تم تحضير نوعين من خليط الوقود الحيوي, و بحيث يمثل الخليط الاول الوقود الحيوي 20% و 80% من الديزل, والثاني عبارة عن 20% من الوقود الحيوي و 60% من الديزل و 20% من الإيثانول و تمت مقارنتة بالمعيار الأمريكي رقم D7467. و جدد ان الخليطين مستوفيين للمواصفات القياسية باستثناء اختبار اللزوجة ، حيث وجد ان درجة اللزوجة للوقود الحيوي تساوي 17.37 درجة مئوية و هي اعلى من المعيار الأمريكي. و كانت قيمة الرقم الحمضي 0.6 mg/KOH

## **Table of Contents**

Contents	Page No.
الإستهلال	I
Dedication	II
Acknowledgment	III
Abstract	IV
المستخلص	V
Table of Contents	VI- XI
List of Tables	XII
List of Figures	XIII
List of Abbreviations	XIV-XV
Chapter One:	
Introduction and Literature Review	
1.1. Energy Definition and Types and Transformation	1
1.1.1 Renewable Energy Resources	2
1.1.2 Heliacal Power	3
1.2 Solar versus Wind	3
1.2.1 Solar versus Hydro	3
1.2.2 Solar versus Biomass	4
1.3 Hydroelectricity	5
1.4 Wind Power	5
1.4.1 Impacts of Wind Power	6
1.5 Biomass Power	8
1.5.1 Impacts of Biomass	9
1.6 Gas emission environmental effects of greenhouse gas	9
emissions	
1.7 Heating and Cooking Emissions	10
1.8 Emission Control System	12

1.8.1 Power systems emission	
1.9 Scientific classification of the <i>Ricinus communis</i>	14
1.10 Description of the <i>Ricinus</i> plant	15
1.11 Benefits and Uses of <i>Ricinus Communis</i> Oil in health.	18
1.12 Environmental Effects of Renewable Energy Technologies	18
1.12.1 Impacts of Solar Power	18
1.12.2 Impacts of Geothermal Energy	19
1.12.3 Impacts of Hydroelectric Power	21
1.13 Biodiesel	21
1.14 Biodiesel production	23
1.15 Trans-esterification reaction	23
1.16 Conventional Transesterification techniques	27
1.16.1 Alkaline transesterification	27
1.16.2 Acid Tran's esterification	28
1.16.3 Trans esterification in supercritical alcohol	28
1.16.4 Water activity	29
1.16.5 Effect of temperature	30
1.17 Biodiesel proportion	31
1.17.1 Density	32
1.17.2 Cloud Point	32
1.17.3 Pour Point	32
1.17.4 Cold filter plugging point	32
1.17.5 Acid number	32
1.17.6 Kinematic Viscosity	33
1.17.7 Density	33
1.17.8 Cloud Point	33

1.17.9 Pour Point	33
1.17.10 Cold filter plugging point	33
1.17.11 Acid Number	34
1.17.12 Calorific Value	34
1.17.13 Water content	34
1.17.14 Color Test	34
1.17.15 Cetane Number	35
1.17.16 Flash Point	35
1.17.17 Copper Strip Corrosion	35
1.17.18 Sulfated Ash	35
1.18 Oil Extraction Techniques	37
1.18.1 Soxhlet extraction	37
1.18.2 Inductively Coupled Plasma Spectroscopy (ICP)	38
1.18.3 Basic principle of ICP	40
1.18.4 Applications of ICP	41
1.18.5 Advantages and Disadvantages of Using ICP	42
1.19 Fourier Transform Infrared Spectroscopy (FTIR)	42
1.20 Gas chromatography- Mass spectroscopy (GC-MS)	44
1.20.1 Gas Supply	44
1.20.2 Injector	44
1.20.3 Column	45
1.20.4 Oven	45
1.20.5 Mass Spectrometer	45
1.20.6 Mass Analyzer	45
1.20.7 Vacuum System	45
1.20.8 Detector	46

1.21 Objectives	46
CHAPTER TWO:	
MATERIALS AND METHODS	
2.1 MATERIIALS	47
2.1.1 Ricinus Communis Seeds collection	47
2.1.2 Reagents and Chemicals	47
2.2 Oil extraction	47
2.2.1 Chemical Extraction	47
2.2.1.1 Cold Solvent Extraction	48
2.2.1.2 Soxidet Extraction Method	48
2.2.2 Mechanical Pressing	48
2.3. Physical and Chemical properties of <i>Ricinus Communis</i> crude oil	49
2.3.1 Physical Properties	49
2.3.2 Chemical Properties	50
2.3.2.1 Total Acid Number	50
2.3.2.2 Free Fatty Acid	51
2.3.2.3 Iodine Value	51
2.3.2.4 Saponification Number	52
2.3.2.5 Peroxide Value	52
2.3.2.6 Calorific Value	53
2.3.2.7 Phosphorus and Elemental Analysis	53
2.4 Fatty Add profile of <i>Ricinus Communis</i> erode oil	54
2.5 Ricinus Communis Biodiesel Production	55
2.5.1 Acid catalyzed esterification process	55

2.5.2 Alkaline catalyzed <i>transeseification</i>	
2.6 Identification of <i>Ricinus Communis</i> methyl ester	
2.6.1 Fourier Transforms Infrared Spectroscopy FTIR	
2.6.2 Gas Chromatography-Mass Spectrometry (GC-MS)	
2.7 Biodiesel blending	
2.8 Physical and chemical properties of <i>Ricinus Communis</i> Biodiesel, , fossil diesel and their blends	
2.8.1 Fossil diesel and B 100 characterization	
2.8.1.1 Physical and chemical properties	57
2.8.2 Biodiesel Blends Characterization	
2.8.2.1 Physical and Chemical Properties	58
Chapter Three:	
Results and Discussions	
3.1 Oil Extraction	59
3.2 Physical and chemical properties of caster crude oil	59
3.3 Fatty acid profile of <i>Ricinus Communis</i> oil	61
3.3 Fourier transform infrared spectroscopy	63
3.4 Ricinus Communis Biodiesel Production	
3.5 Ester content using Gas chromatography-mass spectrometry (Gc-Ms)	
3.6 Physical and Chemical Properties of <i>Ricinus Communis</i> Biodiesel	66

3.7 Physical and Chemical Properties of diesel	68
3.8 Physical and Chemical of Biodiesel Blends	69
3.9 Conclusions	71
3.10 Recommendations	71
Reference	72-91

## **List of Tables**

Table	Table Contents	Page
No.		No.
1.1	Top ten Ricinus Communis oil seed producers Zhang Y, Dube	17
1.2	ASTM D 675145C specification table	32
2.1	Standard methods and Instruments used for physical	49
	properties Ricinus communis crude oil	
2.2	Standard methods used for chemical properties of <i>Ricinus</i>	50
	Communis crude oil	
2.3	Diesel-Biodiesel-Ethanol blends ratio	57
2.4	Methods and Instruments used for physical and chemical	58
	properties	
3.1	Oil yield form caster seed 5 using different extraction	59
	techniques.	
3.2	Physical and chemical properties of caster crude oil	59
3.3	4.3 Elements content of <i>Ricinus Communis</i> crude oil	60
3.4	Display the fatty acids composition of crude Ricinus	62
	communis oil	
3.5	Ester content of Ricinus Communis Biodiesel	65
3.6	Physical and chemical properties of <i>Ricinus Communis</i>	66
	methyl ester	
3.7	Physical and chemical properties of <i>Ricinus Communis</i>	68
	biodiesel	
3.8	Physical and Chemical properties of Diesel	69
3.9	Physical and Chemical properties of Ricinus communis	70
	biodiesel and blends	

## **List of Figures**

Figure No.	Figures Contents	Page
		No.
1.1	Energy Transformation	1
1.2	Global Renewable Power Capacity	2
1.3	Types of biomass source	8
1.4	Leaves and flowers (male flowers on top	14
1.5	Green variant after b looming, with developing seed a capsules	15
1.6	<ul><li>(a) Ricinus Communis oil plant Ricinus communis</li><li>(b) Ricinus Communis oil</li></ul>	15
1.7	Distribution Maps of Ricinus Communis Plant	16
2.1	Agilent ICP-OES 5110	54
2.2	Schematic diagram G-C MS	56
3.1	GC Spectrum of Ricinus communis seed oil	63
3.2	F TIR spectrum of Ricinus Communis oil	63
3.3	GC Mas spectrum of BC	65

#### **List or Abbreviations**

ASTM American Standard for Testing And Materials

EN European Standards

AOCS American Oil Chemist Society

GWh Glossary Gigawatt hours

GHGs Greenhouse Cases

IGCC Integrated Gasification Communed Cycle

CSP Concentrating Solar Plant

ELCA Energy Life Cycle Analysis

NER Net Energy Ratio

FAME Fatty Acid Methyl Ester

FFA Free Fatty Acid

CP Cloud Point

PP Pour Point

CFPP Cold Filter Plugging Point

CSP Communicating sequential processes.

TGA Terrmo-Gravimetric Analysis

ICP Inductively Coupled Plasma Spectroscopy

FTIRS Fourier-Transform Infrared Spectroscopy

GC-MS Gas Chromatography- Mass Spectroscopy

DCN Derivative Cetane Number

ID Ignition Delay

IQT Ignition Quality Tester

TIRS Toxics Release Inventory System

# CHAPTER ONE INTRODUCTION AND LITERATURE REVIEW

## CHAPTER TWO MATERIALS AND METHODS

## **CHAPTER THREE**

**Results and Discussions** 

