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Rheological and physicochemical analysis of non- edible oils used for biodiesel production

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**RHEOLOGICAL AND PHYSICOCHEMICAL ANALYSIS OF NON-
EDIBLE OILS USED FOR BIODIESEL PRODUCTION**

Francisca Zakaria

**A Dissertation Submitted in Partial Fulfilment of the Requirements for the Degree of
Masters of Science in Sustainable Energy Science and Engineering of the Nelson
Mandela African Institution of Science and Technology**

Arusha, Tanzania

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ABSTRACT

The knowledge of the physicochemical and rheological flow characteristics of biodiesel feedstock is used for quality control in production, storage and transportation processes. Moreover, the knowledge is applied in redesigning and optimizing facilities that can be used for manufacturing, storage and transportation of the fuel. Non-edible oils analysed in the present study were Cashew Nut Shell Liquid (CNSL), Castor Oil (CO), croton *megalocarpus* oil (CMO), podocarpus *usambarensis* oil (PUO) and *Thevetia peruviana* oil (TPO). Free fatty acid, acid value, saponification value, peroxide value, iodine value, specific gravity and moisture content were determined by following the recommended Association of Official Analytical Chemists (AOAC) methods of analysing physicochemical characteristics. Physicochemical results show that all the selected non-edible vegetable oils with an exception of CNSL, do not require any further treatment. However, due to high Free Fatty Acid (FFA) content in CNSL, direct biodiesel production from the feedstock would be a challenge hence further treatment to reduce the acidity of the feedstock is essential. Rheological flow parameters were analysed using VT-550 Thermo Haake Viscotester monitored by a Rheowin Job manager. The pre-set parameters in the Viscotester were the shear rate and temperature. The shear rate was increased uniformly from 5 s^{-1} – 100 s^{-1} in 60 seconds at 30-60°C. Regardless of the analysis temperature, all oils exhibited Newtonian flow behavior, indicating that the viscosity of the oil remained uniform at a constant temperature. The experimental data were fitted into rheological models: Newton, Bingham, Oswald de Waele (power-law), and Herschel Bulkley using Rheowin data evaluation software. The results revealed that Newton and Oswald de Waele models were appropriate to represent the flow behaviour of the oils with the fit of $R^2 \geq 0.990$.

DECLARATION

I, Francisca Zakaria, do hereby declare to the Senate of the Nelson Mandela African Institution of Science and Technology that this dissertation is my original work and that it has neither been submitted nor being concurrently submitted for a degree award in any other institution.

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CERTIFICATION

The undersigned certify that, they have read and hereby recommend for acceptance by the Nelson Mandela African Institution of Science and Technology a dissertation titled ***“Rheological and physicochemical analysis of non-edible oils used for biodiesel production”*** in partial fulfilment of the requirements for the degree of Masters of Science in Sustainable Energy Science and Engineering of the Nelson Mandela African Institution of Science and Technology.

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DEDICATION

I dedicate this work to my parents Zacharia Malley and Teckla Tlatlaa Garday, as well as my brothers John Malley, James Malley, Joram Malley and Jeremia Malley.

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LIST OF ABBREVIATIONS AND SYMBOLS

CaCO ₃	Calcium Carbonate
CaO	Calcium Oxide
CAS	Chemical Abstract Service
CAS RN	Chemical Abstract Service Registry Number
CCMS	Concentric Cylinder Measuring System
CFPP	Cold Filler Plugging Point
CMO	Croton <i>Megalocarpus</i> Oil
CNSL	Cashew Nut Shell Liquid
CO	Carbon Monoxide
CO	Castor Oil
CO ₂	Carbon dioxide
ECAT	Exodus Chemicals and Apparatus Trading
EIA	Energy Information Administration
EU	European Union
FAO	Food and Agriculture organization
FFA	Free Fatty Acids
GDP	Gross Domestic Product
GHG	Greenhouse gases
K	Consistency index
KOH/NaOH	Potassium Hydroxide/Sodium Hydroxide
LCSF	Long Chain Saturated Factor
n	Behaviour index
NM-AIST	The Nelson Mandela African Institution of Science and Technology

NO ₂	Nitron Oxide
PUO	Podocarpus <i>Usambarensis</i> Oil
SDG	Sustainable Development Goals
SO ₂	Sulfur Dioxide
TFSA	Tanzania Forest Service Agency
TPO	Thevetia Peruviana Oil
UNEP	United Nations Environment Programme
US EPA	United States Environmental Protection Agency
USA	United States of America
ZnO	Zinc Oxide
γ	Shear rate (s^{-1})
η	Dynamic Viscosity (mPas)
τ	Shear stress (Pa)

CHAPTER ONE

INTRODUCTION

1.1 Background of the Problem

For the past two decades the global energy demand has increased by 60%, surpassing the estimated increase of 40-50% (Gielen *et al.*, 2019). Energy demand is projected to increase by 85% in 2050 (Frei *et al.*, 2013; Mohn, 2020). Currently, 84% of the total energy consumed is derived from conventional sources of energy (Looney, 2020). However, the traditional energy sources are non-renewable and are major source of greenhouse gas (GHG) sources contributing to 35% of GHG emissions (UNEP, 2020), have triggered the pursuit for alternative fuel sources to ensure sustainability and energy security. In 2020, it was reported that 21% of the energy consumed globally is generated from renewable energy sources and an increment to 42% is expected by 2050 (EIA, 2018, 2021). The rise of using renewables is associated with the decline of using conventional energy sources which is projected to fall by 45% in 2050 (Gielen *et al.*, 2019; Nyquist, 2016).

Among other renewable sources of energy, biofuels depict a huge potential to be used as an alternative source of energy and are projected to contribute by 29% of the total energy that will be consumed in the year 2050 (Masi *et al.*, 2021; Schiffer *et al.*, 2018). Biodiesel is a biofuel used as a substitute to fossil diesel. Biodiesel is a significantly promising energy source because of its non-toxicity and biodegradability nature with little to no emissions of GHG. In 2019, the global production of biodiesel increased by 19% (Bockey, 2019) and was used as a 5% blend (B5) to mineral diesel in Europe and USA. The intention is to reduce the GHG emissions to counteract the global climatic catastrophe. Furthermore, Biodiesel is a legally fully registered fuel and blend by US Environmental Protection Agency (Abomohra *et al.*, 2020) ensuring that exhaust emissions produced by biodiesel present no risk to the environment and biodiesel users.

Biodiesel is produced by the trans-esterification process which converts oils and fats into biodiesel (methyl esters) and glycerine. Biodiesel's characteristics depend on the physicochemical and rheological characteristics of the feedstock used during the manufacturing process (Abomohra *et al.*, 2020; Huang *et al.*, 2012). Furthermore, the manufacturing process of biodiesel fundamentally depends on the chemical, structural and flow properties of the vegetable feedstock oil used. Characterisation of the feedstock prior to the production process is therefore very important (Tchameni *et al.*, 2019). Since 95% of biodiesel

produced is from edible vegetable oils (Demirbas *et al.*, 2016; Elgharbawy *et al.*, 2021), feedstock characterisation studies are currently, primarily focused on edible oils. However, the sustainability of biodiesel production from edible oils is affected because of the threat posed on food and energy security (Dias *et al.*, 2016; Lin *et al.*, 2011). To resolve the challenge, the use of non-edible vegetable oils was introduced, nevertheless limited studies have been conducted in analysing the properties of the non-edible oils and its implications on biodiesel production. Furthermore, producing biodiesel from non-edible vegetable oils enhances the affordability of the biodiesel by cutting down the production costs by more than 50% (Demirbas *et al.*, 2016; Shaah *et al.*, 2021). Hence, production of biodiesel from non-edible oils is shown to be a sustainable way of preserving both energy security and food security (Elgharbawy *et al.*, 2021). Therefore, this study analysed the rheological and physicochemical characteristics of non-edible oils that can substitute edible oils in biodiesel production.

Rheology is the study of the nature of flow and deformation characteristics exhibited by the materials under external applied stress (Mezger, 2020). It studies the structural changes under stress, strain and time taken for the changes to occur on properties of a material. It provides a relationship between the properties, structure and processing of the materials (Mezger, 2020; Schramm, 1994). Rheology describes the mechanical behaviour of materials as a function of stress, strain, temperature and pressure. Also, it provides data that is used in developing suitable manufacturing processes and appropriate storage to ensure the stability of the analysed materials (Domínguez *et al.*, 2019). Moreover, the derived rheological data is necessary to maintain the viscosity of the produced biodiesel. Rheological analysis of the feedstock used for biodiesel production is of significance since it analyses the degree of deformation in the molecular structure of the feedstock (Paul *et al.*, 2021). Rheological results are used to predict the biodiesel's flow behaviour as the feedstock's flow behaviour influences it. Additionally, the observed flow properties of the feedstock control specifications of the manufacturing, storage and transportation facilities designs. Fuel quality control and management depend on its structural and molecular sensitivity to the applied external stress. Molecular sensitivity refers to the microstructural changes that are caused by breaking and building up of structures of the oils resulting from the external agents causing flow in this case the stress from shearing and/or temperature changes (Song *et al.*, 2021). Inducing stress to the feedstock oils at different temperatures defines the variation of the microstructural blocks which determines the stability and quality of the feedstock during the conversion process (RheoSense Inc, 2015). Also, the extracted rheological data can be used for the enhancement of biodiesel to improve its quality

and stability based on the flow behaviour exhibited by the feedstock used. Similarly, physicochemical characteristics of the feedstock play an important role in determining the pathway of biodiesel production. Biodiesel production involves pure chemical reactions in the conversion process, thus physicochemical characteristics portray potential implications of using non-edible oils in the production process (Elgharbawy *et al.*, 2021). Moreover, the physicochemical properties of the feedstock are used for predicting and defining the chemical properties of the produced fuel (Shaah *et al.*, 2021). In this study the selected oils for analysis were cashew nut shell liquid (CNSL), castor oil (CO), croton *megalocarpus* oil (CMO), podocarpus *usambarensis* oil (PUO) and *Thevetia peruviana* oil (TPO). Since the second-generation feedstock (non-edible vegetable oils) possess a chemical composition that differs from that of the first generation feedstock (edible vegetable oils) therefore, this study is significant.

1.2 Statement of the Problem

Biodiesel production from edible vegetable oils poses a threat to food security and contributes to higher prices of biodiesel (Demirbas *et al.*, 2016; Iost *et al.*, 2020). To counteract the challenge non-edible vegetable oils can be used in biodiesel production. The use of non-edible feedstock increases biodiesel's affordability because of the reduced production costs since the costs of purchasing feedstock contribute to 80% of the biodiesel's production costs (Elgharbawy *et al.*, 2021). Moreover, manufacturing biodiesel from non-edible oils is convenient as it increases fuel security without compromising food security. However, the challenge of using non-edible oils for biodiesel production is its chemical composition differs from that of edible oils. Limited number of studies focused on analysis of physicochemical and rheological characteristics of non-edible oils. Feedstock analysis and characterisation is suitable for maintaining the fuel's quality and stability by suggesting, remodelling and designing of appropriate manufacturing, storage and transportation facilities and processes. Therefore, this study will establish the rheological behaviour of non-edible oils and the results will be used to suggest suitable manufacturing, storage, transportation processes and facilities that are cost effective in preservation quality and stability of non-edible feedstock biodiesel.

1.3 Rationale of the Study

Limited studies based on exploration and characterization of second generation biodiesel's feedstock inspired this study. Additionally, analysing the quality of the feedstock prior to

production suggests appropriate measures to be taken during and after fuel's production. Therefore, physicochemical and rheological analysis of non-edible oils was performed to provide the information required for:

- (i) Designing appropriate manufacturing and storage facilities for biodiesel from non-edible oils.
- (ii) Optimising the available facilities' performance to meet the requirements for non-edible oils feedstock.
- (iii) Suggesting appropriate feedstock storage and preservation methods to prevent its degradation.

The outcome of the study will be the increased the affordability of biodiesel as an alternative energy source.

1.4 Objectives of the Study

1.4.1 General Objective

To investigate the rheological and physicochemical properties of non-edible oils used for biodiesel production.

1.4.2 Specific Objectives

The study aimed to achieve the following specific objectives:

- (i) To assess the physicochemical characteristics of selected non-edible vegetable oils.
- (ii) To investigate the rheological characteristics of the selected non-edible vegetable oils at different temperatures.
- (iii) To determine appropriate rheological models that define the flow behaviour of selected non-edible oils.

1.5 Research Questions

The study intended to answer the following questions:

- (i) What are physicochemical properties of non-edible oils?

- (ii) How do fluctuations in temperature affect rheological properties of non-edible oils?
- (iii) What rheological models can appropriately represent shear rate-shear stress of non-edible vegetable oils?

1.6 Significance of the Study

Investigating the rheological and physicochemical characteristics of biodiesel's feedstock aids in identifying suitable vegetable oils for biodiesel production. It enables researchers and industries to evaluate the potential of non-edible oils as viable alternatives to edible oils, reducing the competition for food resources and promoting sustainable energy solutions. The rheological properties of non-edible oils can significantly influence the efficiency of biodiesel production processes, such as transesterification, which converts the oils into biodiesel. Therefore, understanding the rheology of these oils helps in optimizing the process parameters, such as reaction time, temperature, and catalyst concentration, leading to improved conversion rates, reduced energy consumption, and enhanced overall process performance. The physicochemical analysis of non-edible oils provides crucial information about their composition, including free fatty acid content, moisture content, density, viscosity, and oxidative stability. These properties directly affect the quality and performance of biodiesel as a fuel. Studying these characteristics helps determine the suitability of non-edible oils for biodiesel production, assess their compliance with fuel standards, and identify potential challenges or modifications required for meeting regulatory requirements.

1.7 Delineation of the Study

The study might be limited by the number of non-edible oils analyzed. The feedstock analyzed may not fully represent the diversity of non-edible oils available, limiting the generalizability of the findings. Non-edible oils can vary widely in their composition, including fatty acid profile, moisture content, and impurities. The study may not capture all possible variations within non-edible oil samples, leading to a limited understanding of the range of physicochemical properties and their impact on biodiesel production.

CHAPTER TWO

LITERATURE REVIEW

2.1 Overview of the Study

Substituting mineral diesel with biodiesel is of important because it diminishes the world's dependence on fossil fuels, provides renewable energy sources and suppressing the amounts of GHG emissions. Biodiesel provides assurance to the transport and industrial sectors which are at least 75% fully operated through the use of fossil diesel (Alleman *et al.*, 2016; IEA, 2020). The quality, properties, oxidative stability and thermal stability of the produced biodiesel, are dependent on the nature of the feedstock used. The chain Length Saturation Factor (LCSF), Free Fatty Acids (FFA), while most studies have not yet established the actual correlation between iodine value and the oxidation stability of the fuel, the relationship between the iodine value and nitrogen oxides emission is well established degree of unsaturation (DU) and saponifiable matter content determine the chemical composition of the methyl ester produced (Domínguez *et al.*, 2019). The characteristics also suggest the pathway to be followed through for the conversion process, favourable manufacturing conditions for maximum conversion efficiency and appropriate facilities to be incorporated in the processes. Even though, biodiesel (B100) can be used in a diesel engine without further modifications of the engine, the physical and chemical characteristics of biodiesel are not exact to that of mineral diesel. The observed differences between diesel CAS Registry Number 68476-34-6 and biodiesel CAS Registry Number 870530-78-2 influence the performance of the engine and the emissions released by the fuels (Bockey, 2019). The ASTM6751 07b and EN14214 established standards for biodiesel to be used in the diesel engine. The comparable standard characteristics of biodiesel and mineral diesel are discussed.

2.2 Biodiesel and Mineral Diesel Characteristics Comparison

2.2.1 Cetane Number

Cetane number expresses the ease of fuel atomisation which determines the quality of combustion. Cetane number in mineral diesel is approximately 46-54 while that of biodiesel is approximately 47-65 (Demirbas, 2009; Karmakar & Halder, 2019). An average cetane number in a fuel has an advantage of cold starting, hence, increasing the engine performance. It also reduces emissions since it does not cause oxidation of sulphur components within the fuel.

However, high values of cetane number can result to fuel knocking. In biodiesel, cetane number is influenced by the high composition of saturated free fatty acids in the feedstock oils used for biodiesel manufacturing (Islam *et al.*, 2018; Siraj *et al.*, 2017). The ASTM6751-07b and EN14214 biodiesel quality standards of US and EU have set a minimum the 47 and 51 cetane numbers, respectively (Alleman *et al.*, 2016; Goosen *et al.*, 2007). Biodiesel quality standard of other countries incorporating the use of biodiesel in transport and industrial sectors is in the range of 47-51 cetane number.

2.2.2 Cloud Point

It is a temperature at which the fuel begins to gel or crystallize. All fuels operate an engine at a temperature that is above their cloud point. Operating an engine below cloud point causes fuel filters to plug and fuel lines to clog, which stops the engine. Biodiesel possesses a higher cloud point (-3 - -20°C) compared to fossil diesel (-40 - -70°C). European countries and the USA use biodiesel as a blend of fossil diesel (Alleman *et al.*, 2016; Islam *et al.*, 2018) since biodiesel has a high cloud point and in extreme coldness biodiesel could completely freeze. Nonetheless, cold flow additives are used to improve biodiesel's cloud point by inhibiting crystals formation. The observed high cloud point in biodiesel results to high Cold Filter Plugging Point (CFPP) of the fuel.

2.2.3 Cold Filter Plugging Point

The measured value CFPP is an indicator of the fuel's filterability at low temperatures. While petroleum diesel CFPP is -70 to -40°C that of biodiesel is -20 to 0°C , implying that additives that can diminish the CFPP are essential for biodiesel to be used in extreme winter (Dunn & Moser, 2005; Girardi *et al.*, 2021).

2.2.4 Flash Point

Is the lowest temperature that is required to start ignition in an engine. Flashpoint is dependent on the cetane number of the fuel. Therefore, the flashpoint of the produced biodiesel is slightly lower (55°C - 60°C) compared to that of mineral diesel (50°C - 60°C). Since biodiesel possesses a high cetane number, lower ignition temperature is required for fuel atomisation (Silva *et al.*, 2015). As a result, it provides quality combustion with lower energy losses to friction (Siraj *et al.*, 2017).

2.2.5 Kinematic Viscosity

It refers to the resistance of the fluid to flow. Biodiesel has high viscosity of about 3.5-6.0 mm²/s at 40°C in comparison to that of fossil diesel which is about 2.5-5.25 mm²/s at 40°C (Siraj *et al.*, 2017). As of now, high viscosity is still the major drawback of biodiesel in the biodiesel industry. Biodiesel's high viscosity results in degradation of the fuel effortlessly. With time a high viscosity biodiesel is converted to a tar like substance which can potentially clog the engine lines and increase the flash point (Demirbas, 2009; Girardi *et al.*, 2021). Therefore, engines, are designed in such a way that there is a limit for maximum viscosity of a fuel to operate. It is to avoid energy loss during the engine starting process because of an escalated ignition temperature from the fuel's high viscosity (Alleman *et al.*, 2016; Islam *et al.*, 2018).

2.2.6 Specific Gravity

According to ASTM6751 07b and EN14214 standards, the specific gravity of biodiesel is within the range of 820-900 (Aga *et al.*, 2020; Howell, 2007). Conversely, the specific gravity of petroleum diesel is approximately 820-845. High specific gravity of biodiesel implies that extra energy is necessary for fuel combustion to occur as opposed to using fossil diesel (Urbán & Józsa, 2018). The specific gravity of biodiesel is however not among the challenges within the industry since most vegetable feedstock oils have the specific gravity that is in synchronization with the recommended standard values.

2.2.7 Sulphur Content

The maximum sulphur content in biodiesel is 10-15 mg/Kg, fifty times less than sulphur content in mineral diesel 500 mg/Kg (Goosen *et al.*, 2007; Shaah *et al.*, 2021). Biodiesel is a sustainable and environmentally friendly because of the small amount of GHG emissions which include sulphur dioxide (SO₂) released. The sulphur molecules in the fuel are oxidised to form SO₂ during the combustion and exhaustion period. More than 10-15 mg/Kg of sulphur molecules release extra volume of SO₂ to the atmosphere. Hence since biodiesel contains less or equal to 10-15 mg/Kg molecules of sulphur low SO₂ is released. The emissions from biodiesel are considered negligible and do not immensely contribute to global warming (Jeswani *et al.*, 2020).

2.2.8 Carbon Residue

Biodiesel is composed of a fewer number carbon-carbon chains compared to diesel. Therefore, combustion reaction that occurs leads to a small composition of carbon monoxide (CO) and carbon dioxide (CO₂) released. Diesel engines perform with high efficiency and hence need extra energy for its operations compared to other liquid fossil fuels. Sharma and Maréchal (2019) reported that 25-40% of the energy consumed by a diesel engine is directed towards cooling the engine system and exhausting the fuel. The reported data suggests that since diesel engines consume more energy, high quantities of CO and CO₂ are released to the atmosphere. It is expected that incorporating 20% biodiesel (B20) in all the transportation systems that solely depend on diesel can reduce CO and CO₂ emissions by 15.7% every five years (Coronado *et al.*, 2009; Siraj *et al.*, 2017).

2.2.9 Oxidation Stability

Rate of biodiesel oxidation depends on the degree of unsaturation leading to the formation of acids, peroxides and tar. Feedstock oil with high composition of unsaturated carbon bonds within free fatty acid bonds, degrades biodiesel's stability. Currently, researchers are focused on stabilising biodiesel. While the oxidation stability of mineral diesel at 110°C is more than 8 hours, the oxidation stability of biodiesel at the same temperature is about 3-8 hours (Goosen *et al.*, 2007; Longanesi *et al.*, 2022) per ASTM6751 07b and EN14214 standards. Degradation of biodiesel when exposed to air at elevated temperatures results in difficulties in handling and storing the fuel (Alleman *et al.*, 2016). Moreover, the degradation of the oxidative stability is caused by the existing impurities that contaminate the fuel which are but not limited to copper, aluminium and iron (Longanesi *et al.*, 2022). Metal contaminants are present in the metallurgy of production and storage facilities and are transferred to the fuel as a result of FFA corroding the facilities (Pöttsch, 2019; Sarin *et al.*, 2009).

2.3 Feedstock Characteristics Influence on Biodiesel's Properties

Biodiesel is characterised by high density, high viscosity, and lower volatility than mineral diesel, affecting a steady fuel atomisation and exhaustion (Silva *et al.*, 2015). This preceding nature of biodiesel is based on the influence of the feedstock vegetable oils' physical behaviour and chemical composition. The most prominent characteristics that influence major properties of the fuels are free fatty acids, long chain saturated factors and iodine value.

2.3.1 Free Fatty Acids

Categorised into saturated fatty acids (stearic and palmitic), unsaturated fatty acids (Linoleic, linolenic and oleic) and polyunsaturated fatty acids. Vegetable oils have high composition of saturated and unsaturated fatty acid which sums up to almost 80% of the oil composition (Shanthilal & Bhattacharya, 2016). However, the FFA composition is not altered during the trans-esterification of oils to produce biodiesel. As a result saturated fatty acids influence cetane number, cloud point, flash point, kinematic viscosity and oxidation stability, primary determinant factors of the fuels quality (EIA, 2018). Unsaturated fatty acids influence the consistency and stability of biodiesel and polyunsaturated acids, in excess influences the formation of polymers at high temperatures. Also, excess amounts of polyunsaturated acid decreases cetane number, cloud point and oxidation stability of the produced biodiesel (Chuah *et al.*, 2016; Islam *et al.*, 2018). Generally during the production, the presence of FFA in the feedstock can affect the production process by diminishing the conversion efficiency because saponification reaction is influenced (Elgharbawy *et al.*, 2021; Toscano *et al.*, 2012). Saponification reaction is the soap forming reaction brought about by the reaction between methyl esters (biodiesel) and a strong alkali. Additionally, high FFA composition affects post production processes which are essential in maintaining or improving the fuels quality.

2.3.2 Long Chain Saturated Factor

The length of the chain of the feedstock oil determines cetane number, viscosity and iodine value of the produced biodiesel. Longer chains of the feedstock lead to high cetane number and high viscosity of biodiesel and vice versa. It is due to long carbon chains suggest that many carbon molecules are joined together to form the chain. A study by Islam *et al.* (2018) states that vegetable oils have a longer fatty acid chain which leads to the production of biodiesel with high cetane number and viscosity. Moreover, a study by Chuah *et al.* (2016) stated that there is a correlation between LCSF and CFPP i.e. longer chains of the feedstock lead to biodiesel high CFPP which means the fuel has high viscosity, has high ignition temperature and cannot be used in cold areas due to the fact that the freezing point of the fuel is high.

2.3.3 Iodine Value

Islam *et al.* (2018) reported on the iodine value of the feedstock oils seems to be higher up in the range of 69.3-135, as a result of excess amounts of unsaturated fatty acids in non-edible oils. Iodine value determines the degree of unsaturation of the fuel (Islam *et al.*, 2018).

Therefore, feedstock oil's unsaturation mainly determines biodiesel's general stability. Degree of unsaturation means the presence of double bond in the oil feedstock which even after the trans-esterification process yields biodiesel which can easily decompose due to the presence of double bond in the product as well. Decomposition due to the presence of double bond in biodiesel occurs at an exponential rate and takes place really fast leading to the formation of acids and peroxides (Chuah *et al.*, 2016; Islam *et al.*, 2018). Furthermore, iodine value determines the amount of nitrogen oxides (NO and NO₂) emission. While most studies have not yet established the actual correlation between iodine value and the oxidation stability of the fuel, the relationship between the iodine value and nitrogen oxides emission is well established (Neupane, 2022; Pullen & Saeed, 2014). The higher the iodine value means higher nitrogen oxide emission.

2.4 Types of Biodiesel's Feedstock Used in Production

The feedstock used for biodiesel production is characterised into 4 different groups according to FAO as the first, second, third and fourth generation feedstock (Karmakar & Halder, 2019). First generation feedstock are the edible oil crops which were used since the beginning of the industries' production process. To date the biodiesel market is dependent on edible oils such that 95% of the biodiesel produced is from edible oils. The sources include sunflower, soybean, palm. Over the years first generation feedstock was and is still highly discouraged due to the rising impacts on the food market. Still, the first generation feedstock is used by large biodiesel producer countries since the produced biodiesel from the oils is easily maintained over time without occurrence of drastic change. As of now, second generation feedstock is highly encouraged as an alternative to the production of biodiesel (Abomohra *et al.*, 2020; Ikram *et al.*, 2019). Second generation feedstock are non-edible oils such as castor oil, jatropha and croton *Megalocarpus*. This generation of feedstock has advantages over the first generation feedstock. Non-edible oils are composed of over 0.5% of FFA enhancing its unsuitability to be used for human consumption hence, interfere with the food market. The biggest challenge with this generation of feedstock is the unreliability of the materials and the negative influence of their chemical composition on biodiesel quality (Karmakar & Halder, 2019). Nevertheless, it is a generation of feedstock that is considered for future production of biodiesel.

Third generation feedstock is micro algae which is considered be a feedstock that will be profitable for commercial scale production, unlike the first and second generation feedstock (Ikram *et al.*, 2019). Also, there is no competition with food for this generation but still this

generation faces many technological challenges and it still is researched on (Ikram *et al.*, 2019). Fourth generation feedstock intends to trap and store CO₂ from the feedstock so that it is not released to the environment. The feedstock is genetically modified algae species with high capacities of biomass accumulation responsible for biodiesel production through photo-biological solar cells (Abdullah *et al.*, 2019). Studies on the development of the feedstock are still on going. However, The technology is yet to be utilised (Karmakar & Halder, 2019).

2.5 Biodiesel Production from Edible and Non-Edible Oils

World market of biodiesel is dominated by production of biodiesel from edible oils. The production market gravitated towards it because of the reliability and average amount of FFA in edible oils. Biodiesel production and marketing from edible oils started three decades ago in Europe (Germany and France) before it was accepted in other parts of the world. After it was evident that biodiesel had increased the quality of diesel fuel by 7% in European countries, other nations incorporated the use of biodiesel as a blend of diesel. For the past decade, a leading producer of biodiesel worldwide was the United States of America (USA) which contributed to an average of 41% of the total biodiesel produced (Kayode & Hart, 2019). Other major producers were Brazil with a share of about 26%, Indonesia with 4.5%, China with 2.9% and Germany with 2.8%. According to the report by Bockey (2019), biodiesel production has increased by 13% in 2019 to 41.4 billion litres.

The 5 leading countries in the production contributed to 57.3% of the total biodiesel worldwide. These major producers were Indonesia 17%, USA 14%, Brazil 12%, Germany 8% and France 6.3% (Quah *et al.*, 2019). Production of biodiesel is expected to increase globally as a result of new formulated policies which advocate to no taxation of biodiesel, especially in the developed countries. In the USA, it is expected that in 2025 biodiesel production industry will have grown tremendously due to the domination of commercial vehicles are designed to use biodiesel and other biofuels (Alleman *et al.*, 2016). Currently, the diesel market comprises of 10% biodiesel from edible oils, which is mainly used as a blending component for diesel fuel in to improve its biodiesel's quality for diesel engines (Alleman *et al.*, 2016). The biggest biodiesel producing company in the US is Renewable Energy Group producing approximately 701 000 tons of biodiesel annually from six plants (Rianawati *et al.*, 2021).

Africa is viewed to have more potential renewables including biodiesel production. Africa shows excess potential of incorporation biodiesel production from non-edible oils. Currently,

Africa has no large plants that are designed for biodiesel production, but the market is occupied by minor and medium scale producers. All other African countries are still in the initial stages of biodiesel production with the exception of South Africa, Mozambique and Zimbabwe (Gashaw *et al.*, 2015). All major biodiesel plants in Africa use non-edible oils feedstock for biodiesel production. It is because of the extensive availability the second generation feedstock for biodiesel. Africa's major and first ever biodiesel plant is located in Zimbabwe. The plant uses *Jatropha* as the main feedstock among many others. The expectation of the plant is to generate about 100 ML of biodiesel annually and save up to almost 80 million US dollars from the importation of fossil diesel. However the shortage of feedstock resulted to this plant operating at only 5% of its capacity (Gonzales, 2016). In 2007 Mozambique installed its first biodiesel plant in Matola. The plant used *Jatropha* for the production of biodiesel. Mozambique's government intended to farm for *Jatropha* in order to have a reliable supply of raw materials for the plant (Henley, 2014). It was expected that in 2019 a biodiesel plant is to be installed in Johannesburg, South Africa. The plant is to use waste coffee grounds as the main feedstock for production of biodiesel (Rezania *et al.*, 2019). It was estimated that the plant would generate 1.8 million litres of biodiesel. The plant is designed to recycle 75 000 tonnes of coffee waste which when disposed would be toxic and damage the environment. Also, it would lead to saving 476 000 tonnes of CO₂ emissions. The plant is still under construction as of now (Rezania *et al.*, 2019). Other African countries such as Mali, Uganda, Zambia and Tanzania are also now indulging in the production of biodiesel.

Manufacture and commercialisation of biodiesel in Africa contributes to the expansion of different sectors such as industrial sector, business sector and agriculture sector. Therefore, non-edible oils are still not well incorporated in the production industries in the major biodiesel producers' countries. The production of biodiesel from edible oils was however highly questioned from 2007-2008 after the global recession and when the food crisis that was at its peak. To mitigate the concerns brought up, non-edible oils were considered and mentioned to be the future of biodiesel production. However, the production had not immediately started because of the limited production technologies suited for production from non-edible oils. With further research being conducted, the biodiesel industry technologies are continuously evolving, and it is expected non-edible oils with the right policies could completely replace edible oils as the primary biodiesel feedstock.

2.6 Production Technologies

Production technologies of biodiesel have evolved over the years following different researches conducted to improve biodiesel quality. Most of the modern technologies that are expected to incorporate non-edible raw materials in the production processes are still in development stages and expected to be fully utilised in the future. Biodiesel plants operate globally on conventional biodiesel production approaches (Abbaszaadeh *et al.*, 2012; Karmakar & Halder, 2019). A recent study by Supriyanto and co-workers highlighted that the transesterification technique is still the most superior process used in biodiesel production from vegetable oils (Supriyanto *et al.*, 2021). Also, this study emphasised the importance of analysing the physicochemical and rheological characteristics of the vegetable oils used as biodiesel raw materials. It is because feedstock characteristics influence the specifications of the new designs for production processes. Therefore, until the new technologies are mature enough to be used and reliable, commercial biodiesel production will depend on the catalysed conventional approaches of production.

2.7 Challenges in the Biodiesel Industry

The challenges facing the biodiesel industry are divided into three categories which are pre-production challenges, production challenges and post production challenges. Primary pre-production difficulties include a competitive feedstock market and variation of chemical and physical characteristics of the feedstock oils (Anuar & Abdullah, 2016). While an ample amount of research is conducted to determine alternative substances that can be used as biodiesel feedstock, inconsistencies of biodiesel properties occurring as a result of variation of the feedstock's composition are difficult to maintain.

The production process is dragged back by the production facilities compatibility and the moisture content in the feedstock oil. Moisture in the biodiesel's feedstock hydrolyses the fats into acids and peroxides. The moisture content of the feedstock is to be maintained below 1% in the feedstock before production. However, it is a challenge since heating of the feedstock to remove moisture takes place at the temperature 55-70°C (Fregolente *et al.*, 2012; Pöttsch, 2019). At 55-70°C temperature, oxidation of the feedstock can take place since the elevated temperatures enhance the substitution reactions to take place.

Also, physical nature and chemical composition limits the materials that can be used during the production processes. Some cheap metals used to make production plants, such as copper, zinc

or alloys of these metals (brass and bronze) degrade over time upon usage with biodiesel (Severo *et al.*, 2019). Aluminium has shown compatibility with pure biodiesel, however since the transesterification process takes place in the presence of a strong alkali (KOH/NaOH) as a catalyst, it also is discarded as the building material for biodiesel plants. More research is however focused on stainless steel and carbon steel materials to be used for production, storage and transportation of biodiesel (Pötzsch, 2019). Also, challenges are reported on the feedstock's flow behaviour, particularly those of high viscosity. High viscosity feedstock results to major operations difficulties including the mixing process and transfer process through the production tanks (Coronado *et al.*, 2019; Peterson *et al.*, 2002).

Post production facility challenges are similar to production facility challenges. Additionally, the by-product of biodiesel production (glycerol) is currently of little value; hence, since the by-product contributes no returns on investment, the cost of biodiesel spikes.

2.8 Socio-Economic Impacts of Biodiesel Industry

The biodiesel industry contributes to the achievement of Sustainable Development Goals (SDGs). The industry particularly addresses SDG number 1 that emphasises on poverty elimination, SDG number 7 emphasising on availability of sustainable, renewable and clean energy and SDG number 13 emphasising on the climate change action (Ji & Long, 2016).

For SDG 1, the biodiesel industry has created employment opportunities that reduce the unemployment rate hence increasing the number of individuals living above the poverty line. Since, the biodiesel's fundamental raw materials include a variety of vegetable oils, the agriculture sector benefits through the trade created between the energy sector and the agriculture sector. Consequently, the nation's manufacturing and trading biodiesel observe an increase in the GDP per capita. A report by Ditzel *et al.* (2018) stated that in 2017, 6.5 billion dollars from the biodiesel industries contributed the US GDP per capita. The contribution sources included but not limited to the employment opportunities created, labour income, federal taxes, state taxes and local taxes.

The report also analysed the contribution of the industry in achieving SDG 7 and SDG 13. The report stated that the incorporation of biodiesel in the energy sector has increased energy security index and reduced the GHG emissions simultaneously. The GHG emissions were reduced by 14.8 million tons from the use of biodiesel only. Therefore, SDG 7, which encourages the use of clean, renewable energy sources that can replace fossil fuels and SDG

13 which emphasised on limiting the climatic changes from occurring, are both simultaneously being taken care of through the biodiesel industry.

However, biodiesel production in the world market is entirely dependent on edible oils and could potentially result to food crisis in the future. Thus, though biodiesel industry is thriving and contributes to achieving SDG number 7 and SDG number 13, it compromises SDG number 1. Second generation feedstock is therefore essential to making the biodiesel industry a pure advantageous industry in the energy sector and the environment sector without jeopardising other sectors. The second generation feedstock is still under research so that appropriate facilities necessary for undertaking all the involved facilities during production and post production can be designed (Bhuiya *et al.*, 2014).

2.9 The Future of Biodiesel Industry

Up to 2022, 95% of the total produced biodiesel was from edible oils and 5% was from animal fats and non-edible oils (Iost *et al.*, 2020). It is reported that intensive exploration of edible oils for biodiesel production has imposed a threat of deforestation, especially in Asian countries (Indonesia, Malaysia), which are the largest producers of palm oil (Jeswani *et al.*, 2020). Deforestation is the chief concern for environmentalists, stating that biodiesel is rather environmentally unfriendly since deforestation contributes to approximately 10% of global warming (Lawrence *et al.*, 2022). Hence, environmentalists started a movement that is against biodiesel production from palm oil. Palm oil is popular as a biodiesel feedstock in European Union, USA and Indonesia whereas Soybean oil and rape seed oil are popular in North and South America (Afriyanti *et al.*, 2016). In 2018, EU parliament banned the use of palm oil for biodiesel production from 2021 which resulted from the protest against deforestation (Faishal & Tjitrawati, 2020). Therefore, other edible feedstock oils which are rapeseed oil and soy oil were incorporated in biodiesel production plants in the EU.

Second generation feedstock (non-edible oils) is considered to be the most viable option for biodiesel production from now into the future even though until now plants in the major producer countries depend on vegetable oil (Matzenberger *et al.*, 2015). However, as a result, non-edible oil plants are highly encouraged to be grown in inhabited areas where there are wastelands or forest areas. For example, Soapnut is wildy grown in forests of Nepal and *Jatropha* plantations in Mozambique.

In the African continent at the present, non-edible oils are utilised for biodiesel production. Strategies are continuously established to grow the plants which can be used for biodiesel production in the continent without compromising on food. Production of biodiesel within Africa will reduce importation costs of oil from other continents. It also, shall increase the affordability of biodiesel within the continent and generate revenue for exporting. Long term, the plantations will be intentionally developed in order to increase the reliability of the feedstock that will be available without taking into consideration the food market factor.

2.10 Policies to Foster and Regulate the Industry

European countries were the pioneers of implementation of policies that would regulate the utilisation of biodiesel so that the goal of at least 10% of all the transportation fuel in the year 2020 should be from renewable energy sources. In order to reach the goal all taxes on biodiesel were removed from 2003 (Böhringer *et al.*, 2009; Ji & Long, 2016). The policies that were set in European countries were then adopted by other nations including major biodiesel producers USA and Brazil. The policies aimed at the encouragement of biodiesel businesses which were growing at the time.

African countries producers of biodiesel such as South Africa, Mozambique and Zimbabwe also adopted the policies from the Europe. Africa is considered to be the continent where there is a huge potential for renewable energy investment as it has not yet been completely exploited of its sources. Still, policies and regulation in Africa on biodiesel are not clear and on place. Continuous changes are made to the policies however, as other countries South Africa removed all the taxes associated with biodiesel production and trade (Henley & Fundira, 2019). Policies and regulations in Africa are continuously being developed to cater for the needs of the people and develop motivation for the use of biodiesel. Since Africa is viewed as the continent with the highest potential of biofuels worldwide, the biofuel sector in Africa can lead to an increased revenue generation to African countries. Furthermore, the sector can generate business and employment opportunities for Africans. This should influence stable policies and regulations guiding the industry.

2.11 Characterization of Biodiesel Feedstock

Physicochemical and rheological characteristics of the feedstock convey the relationship between the conversion process during the production and the biodiesel produced. Physicochemical characterisation is linked to the influence of physical and chemical

characteristics of the feedstock to that of the produced fuel. Rheological studies establish the relationship between shear stress and shear rate at different pre-set conditions. Rheological properties describe the ease of flow and the influence of the flow on the viscosity of the produced biodiesel in a set provided time. Its chemical composition and physical properties define rheological characteristics of the feedstock. As a result, the produced fuel qualities are as well defined regarding the feedstock's exhibited characteristics. Analysed feedstock oil samples can exhibit different flow properties depending on the temperature and pressure set. Also, the differences in the flow properties can be influenced by the morphological structures of the feedstock.

Therefore, the rheological characterisation of the feedstock determines the biodiesel's thermal stability. A number of rheological studies have been conducted to study the thermal properties of edible oils that are used to produce biodiesel. A study by Silva *et al.* (2015) had evaluated the rheological behaviour of vegetable oils (cotton, canola, sunflower, corn and soybean) at different temperatures. To predict the flow behaviour trend of the feedstock oils at high shear rates, the investigation was based on four rheological models that described the nature of flow exhibited which were Ostwald-de- Waelle, Herschel-Bulkley, Newton and Bingham in which the samples were observed to be Newtonian fluids. Newtonian fluids show a linear shear rate shear stress relationship. As the temperature of the oil increased, its viscosity reduced (Silva *et al.*, 2015). Edible oils have low content of Free Fatty Acids in comparison to non-edible oils resulting to high viscosity on the produced biodiesel from non-edible oils. However, a limited number of studies established the flow properties that are exhibited from non-edible oils feedstock.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials and Chemicals

Cashew nut liquid shell (CNSL) was obtained from Korosho Africa Limited, a local cashew nut processing factory in Tunduru district, Southern East Tanzania. Castor Oil (CO) was from the local small seed processing industry in Dodoma, central Tanzania. Cashew nut liquid shell (CNSL) was obtained from Korosho Africa Limited, a local cashew nut processing factory in Tunduru district, Southern East Tanzania. Castor Oil (CO) was obtained from the local small seed processing industry in Dodoma, central Tanzania. Croton *Megalocarpus* and Podocarpus *Usambarensis* seeds were purchased from Tanzania Forest Service Agency (TFSA). The seeds were collected from Usambara forests in Lushoto, Tanga in the Northern East part of Tanzania. *Thevetia Peruviana* seeds were collected from the outskirts of Arusha and Kilimanjaro regions in the northern part of Tanzania.

The selection of the analysed oils was dependent on oil yield (with oil yield limit > 20%), seeds maturity index (time taken for the seeds from sprouting to maturity before harvest where the time limit is < 15 years), the reproduction term (the life span) of the seed-bearing plant. Factors that were taken into consideration in selecting appropriate non-edible feedstock oils that can be used in biodiesel production at the moment and in the future are summarized in Table 1.

Table 1: Feedstock oil selection factors

Feedstock oil	Oil Yields (%)	Maturity Index (months)	Life span (years)	Citation
CNSL	20-30	6 – 18	50	Gandhi <i>et al.</i> (2012) and Kyei <i>et al.</i> (2019)
CO	28-59	6 – 18	4	Román-Figueroa <i>et al.</i> (2020)
CMO	35-45	48	45	Aga <i>et al.</i> (2020) and Aliyu <i>et al.</i> (2010)
PUO	50-67	180	75	Minzangi <i>et al.</i> (2011)
TPO	50-67	6 – 18	45	Bora <i>et al.</i> (2014)

Figure 1 shows the selected seeds and cashew nut shells that were used for oil extraction. Except for Castor seeds and Cashew nut shells, the figure shows other seeds prior to the shelling process which separated kernels and the seeds coats.



Figure 1: The seeds from which the feedstock oil was extracted (A) Cashew nut shells, (B) Castor seeds, (C) *Croton Megalocarpus* seeds, (D) *Podocarpus Usambarensis* seeds and (E) *Thevetia Peruviana* seeds

Reagents used as solvents for oil extraction were analytical grade petroleum ether and chloroform purity 99%. For physicochemical characterisation of the samples, chemicals used were analytical grade petroleum ether, ethanol, POP indicator, potassium hydroxide, hydrochloric acid, glacial acetic acid, chloroform, potassium iodide, starch indicator, sodium thiosulfate, carbon tetrachloride, and Wiji's solution. The reagents were bought from Exodus Chemicals and Apparatus Trading (ECAT) in Arusha CBD.

3.2 Sample Preparation

Castor oil and cashew nut shell liquid was analysed without any further treatment. Croton *Megalocarpus*, Podocarpus *Usambarensis* and *Thevetia Peruviana* seeds were separated from the kernels using mechanical methods such as hammering. The kernels were then washed with water to remove fine dust accumulated on the seeds during the shelling process. To ensure the moisture content of the seeds is extremely reduced before the extraction process, the seeds were sun dried for 3 days, for 4 hours a day, consecutively. After drying, the seeds were crushed using a blender (Dessini, model: DS-138, 450W) for 3-5 seconds to obtain particles that were of approximately 2 mm-5 mm diameter. The seeds were blended to increase the surface area for oil absorption during the extraction process.

3.2.1 Oil Extraction

Oil extraction from the seeds was done by employing the solvent extraction method as described in the literature (Kibazohi & Sangwan, 2011; Temitayo, 2017). Solvent selection for oil extraction depends on the affinity of the solvent for specific oil (Dhoot *et al.*, 2011; Suwari *et al.*, 2017). Hence, petroleum ether with CAS Registry Number 8032-32-4 was used to extract croton *megalocarpus* oil and podocarpus *usambarensis* oil from the seeds (Aga *et al.*, 2020; Minzangi *et al.*, 2011). Chloroform with CAS Registry Number 67-66-3 was used to extract *Thevetia Peruviana* oil from the seeds (Yadav *et al.*, 2016). Selection of the solvent was in accordance with the research done in the identification of the oil affinity for various solvents (Kessler *et al.*, 1985; Temitayo, 2017).

The crushed seeds were placed in a black plastic air tight container in which the respective solvent was added and covered to allow the extraction process to occur. The lid of the bucket was completely tightened to ensure no infiltration of the solvent to the atmosphere. For approximately 1 kg of the crushed seeds 250 ml of the solvent was used. The oil from the seeds was allowed to dissolve in the solvent for 12 hours prior to filtration to ensure maximum amount of oil is extracted from the seeds (Gandhi *et al.*, 2012; Kessler *et al.*, 1985; Suwari *et al.*, 2017). After filtration the oil-solvent mixture was placed in a clean container and using a water bath the solvent was evaporated at a temperature range of 40-50°C from the mixture for approximately 2 hours, until no more bubbles of the solvent exiting the mixture were observed which ensured full evaporation of the solvent. The water bath was used to ensure the safety during the experiments, both petroleum ether and chloroform are highly flammable and hence

would catch fire easily when used with the direct flame during the evaporation process. The extracted oil was analysed with no further treatment. A series of steps that were carried out during the oil extraction process from their respective bearing seeds are as summarized in Fig. 2.

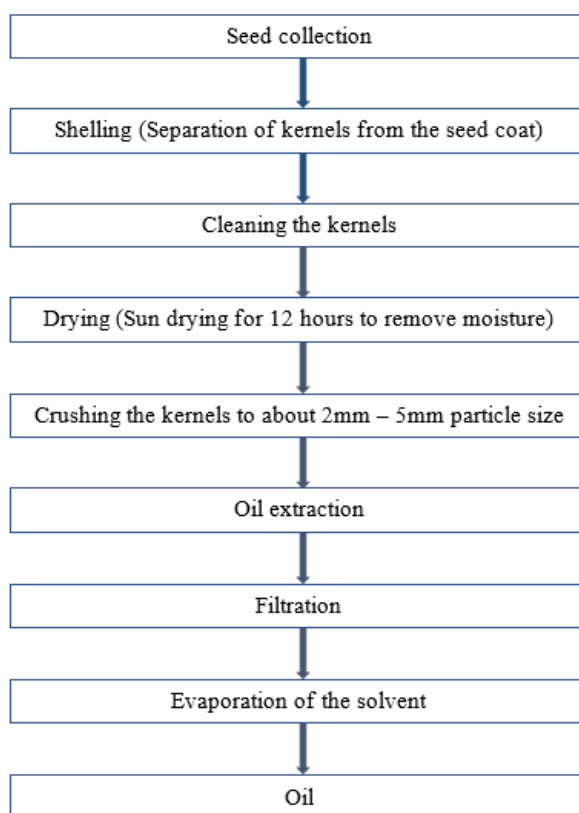


Figure 2: A series of steps followed in the feedstock oil extraction process

3.3 Physicochemical Characterization of the Samples

Physicochemical parameters employed Association of Official Analytical Chemists (AOAC) International standard analysis methods in the characterisation of oil samples. Detailed procedures for each parameter analysed are expressed as follows:

(i) Acid value

A mixture of 60 ml petroleum ether/ethanol 2/1, was poured into a dry 250 ml conical flask. Two grams (2g) of the sample oil was added to the conical flask while continuously stirring. To the oil/solvent mixture, two drops of POP indicator were added, and the mixture was then titrated against 0.1N of potassium hydroxide until a pink colour that persisted for 5 minutes

was observed. Acid value was calculated by the equations 1 and 2 (Bouaid *et al.*, 2016; Cunniff & Washington, 1997):

$$AV = \frac{56.1 \times V \times N}{m} \quad (1)$$

V = Volume of the titrant (KOH) in ml required for neutralisation

N = Normality of KOH

m = mass of the oil sample in g

(ii) Free Fatty acid

Acid value obtained was used for free fatty acid (FFA) determination using the equation:

$$FFA = \frac{AV}{2} \quad (2)$$

(iii) Saponification Value

A mixture of 2 g of the sample oil and 25 ml of freshly prepared 1.0 M alcoholic KOH (ethanol potassium hydroxide) were placed in a dry 100 ml volumetric flask. The flask was then attached to a condenser and the solution was heated to its boiling point for 30 minutes in order to allow completion of saponification reaction. Saponified solution mixture was allowed to cool, and 3 drops of POP indicator were added and was titrated against 0.5 N HCl until the endpoint was reached. Similar procedures were employed for determination of a blank value. Saponification value was calculated using the expression 3 (Cunniff & Washington, 1997; Ifijen & Nkwor, 2020):

$$SV = \frac{56.1 \times N \times (V_B - V_S)}{m} \quad (3)$$

N = Normality of HCl

V_B = Volume of HCl used in a blank test

V_S = Volume of HCl used in the actual test

m = mass of the oil sample in g

(iv) Peroxide Value

To a dry Erlenmeyer flask 5 g of oil sample was added to be dissolved by 30 ml of solvent mixture, glacial acetic acid/chloroform, 3/2, v/v mixture. The 0.5 ml of freshly prepared potassium iodide was added to the flask as well and was stirred for 1 minute to form a homogenous mixture. To the mixture, 15 ml of distilled water was added and stirred for 1 minute. The mixture was then titrated against 0.01 N of sodium thiosulphate using starch indicator until the endpoint was reached and formula 4 was used in determination of the peroxide value (Cunniff & Washington, 1997; Ifijen & Nkwor, 2020).

$$PV = \frac{V \times N \times 1000}{m} \quad (4)$$

V = Volume of sodium thiosulphate used

N = Normality of sodium thiosulphate

m = mass of the oil sample in g

(v) Iodine value

To a glass-stopper of 250 ml capacity flask, 10 ml of carbon tetrachloride and 2 g of sample oil were poured and mixed well. To a mixture, 20 ml of Wiji's solution was added, stopper was inserted, and the mixture was put in darkness for 30 minutes. The 100 ml distilled water was added into the sample flask while continuously stirring. To the sample mixture, 15 ml of freshly prepared 10% potassium iodide was added, followed by titration of the mixture against 0.1 N of sodium thiosulphate solution using 1.5 ml of starch indicator until the endpoint was achieved. The same procedures were carried out for the blank sample simultaneously and the iodine value was calculated from equation 5 (Cunniff & Washington, 1997; Suwari *et al.*, 2017).

$$IV = \frac{12.69 \times (V_B - V_S) \times N}{m} \quad (5)$$

N = Normality of sodium thiosulphate

V_B = Volume of sodium thiosulphate used in a blank test

V_s = Volume of sodium thiosulphate used in the actual test

m = mass of the oil sample in g

(vi) Specific gravity

The weight of a cleaned and dry density bottle was taken, W_0 . The density bottle was then filled with oil samples and was reweighed to give weight W_1 . The same procedure was used to obtain W_2 , weight of distilled water in the density bottle (Suwari *et al.*, 2017). Equation 6 was used in calculation of the specific gravity values of all the samples.

$$SG = \frac{W_1 - W_0}{W_2 - W_0} \quad (6)$$

W_0 = weight of an empty bottle

W_1 = weight of an empty bottle + weight of sample oil

W_2 = weight of an empty bottle + weight of distilled water

(vii) Oil yield

Mass of the dry seeds ready for oil extraction was recorded as m_0 . After extraction the mass of oil extracted was obtained and recorded as m_1 (Román-Figueroa *et al.*, 2020). Using Equation 7 the percentage oil yield from the samples was obtained.

$$\text{Oil yield (\%)} = \frac{m_1}{m_0} \times 100 \quad (7)$$

m_0 = Mass of the seeds before extraction

m_1 = Mass of the extracted oil

(viii) Moisture content

In a 250 ml capacity Pyrex beaker, the sample oil of 50 g was poured and was placed inside an oven at a temperature of 45°C for 3 hours ensuring proper expelling of the moisture from the samples. After each hour, the sample was taken out of the oven, was allowed to cool and was

weighed. Weighing sample oil was repeated until there were no any further changes in its weight, insinuating that all the moisture in the sample was evaporated (Omari *et al.*, 2015). Equation 8 was used for determining the moisture content in the samples:

$$\text{Moisture content (\%)} = \frac{m_0 - m_1}{m_0} \times 100 \quad (8)$$

m_0 = Weight of sample oil + Moisture

m_1 = Weight of dry sample oil

Table 2 presents the integrated method of analysis identification number assigned by AOAC international analysis standard methods and the primary principle of analysis followed.

Table 2: Physicochemical characterisation techniques

Parameter	Method	Principle
Oil yield	-	Gravimetry
FFA	AOAC 940.28	Titrimetry
AV	AOAC 940.28	Titrimetry
SV	AOAC 920.160	Titrimetry
PV	AOAC 965.33	Titrimetry
IV	AOAC 920.159	Wiji's titrimetry
SG	AOAC 920.212	Pycnometry
Moisture content	AOAC 930.15	Gravimetry

FFA= Free Fatty Acid, AV= Acid Value, SV= Saponification Value, PV= Peroxide Value, IV= Iodine value, and SG= Specific Gravity

3.4 Investigation of Rheological Characteristics of Oils

Rheological flow properties of the samples were analysed using a shear rheometer, Haake Viscotester, model VT 550, Karlsruhe, Germany. This research used a Concentric Cylinder Measuring System (CC MS) employing Searle operating method for the experiments. Medium viscosity of the samples analysed influenced the selection of Searle's operational method (Mezger, 2020; Schramm, 1994).

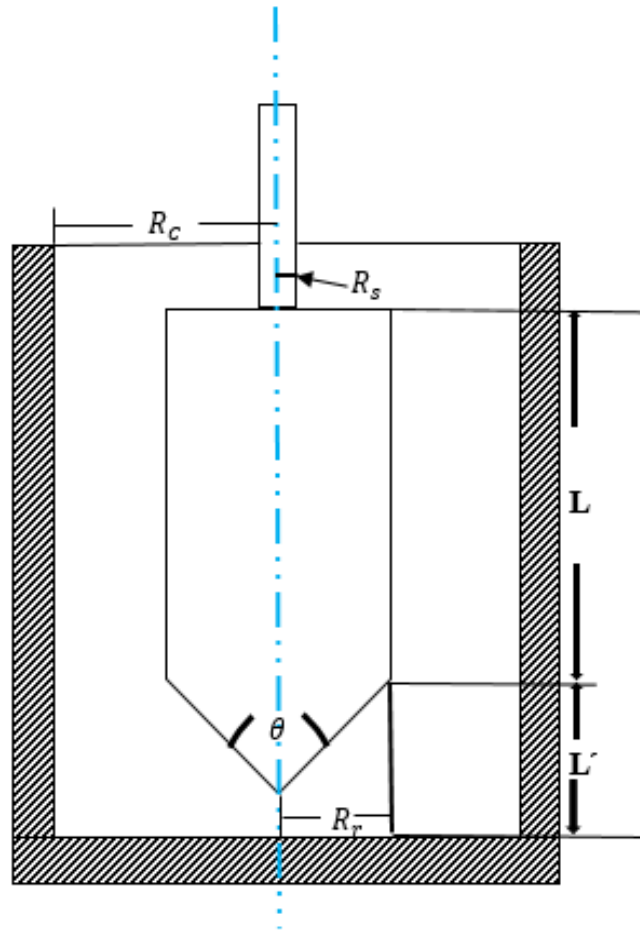


Figure 3: Haake rotational rheometer setup for the experiments

The measuring system entailed a stationary outer cylinder mounted on the same axis as the rotating inner cylinder (sensor system). The gap between the outer and inner cylinder was filled with the sample to be analysed, as shown in Fig. 3. To ensure uniform stress distribution on the sample between the concentric cylinders, the degree of heterogeneity of stresses ($\varepsilon = \frac{R_C}{R_r}$) for the setup was 1.084. The literature recommended the ratio of the outer cylinder to that of the inner cylinder to at least be a unit to get accurate measurements (Mezger, 2012; Schramm, 1994).

As the inner cylinder was in rotation, adequate torque to subdue the sample's viscosity, posing resistance to the induced motion, was measured. Rheological flow parameters analysed in concurrence with the system fixtures and speed of inner cylinder (ω) expressed as shown in equations 9, 10 and 11:

$$\gamma = \frac{2\omega R_c^2}{R_r^2 - R_c^2} \quad (9)$$

$$\tau = \frac{M}{2\pi R_r^2 L} \quad (10)$$

$$\eta = \frac{\tau}{\gamma} \quad (11)$$

In equations (9)-(11): γ , τ , η and M represents shear rate (s^{-1}), shear stress (Pa), viscosity (Pas), and the torque measured (Nm), respectively.

System operation throughout the experiments was controlled by Rheowin 3 Job Manager software, version 3.6. The rheometer was operated under controlled shear rate mode (CR), and the rheological characteristics of the samples were analysed at 30°C, 40°C, 50°C, and 60°C, the variation is based on the production temperature range applied in the industries. The temperature was measured by a thermostatic sensor (Pt 100) coupled with the device. For each experiment conducted, the shear rate would increase from 5 s^{-1} to 100 s^{-1} for 360 s. To ensure the precision of the results, experiments were carried out in triplicates using a fresh sample for each experiment. Rheological parameters were computed using the mean values of the replicates on the Data manager software. The research focused on the relationship exhibited by the shear stress and viscosity parameters, with temperature variation at different shear rates.

3.5 Determination of Rheological Models Describing the Flow Behaviour

Raw data presented in rheograms was fitted into four rheological models in a data managing software to determine the model(s) that describes the rheological properties of the samples. Models selected to investigate the correlation among the parameters were Bingham, Herschel-Bulkley, Newton, and Oswald De Waele, also known as the Power-law model. On the account of time independent characteristic observed from the studied samples the rheological models were selected to represent the flow behaviour of the samples (Mezger, 2020; Shanthilal & Bhattacharya, 2016). Mathematical expressions 12-15 display the parameters of the respective rheological models are:

Bingham

$$\tau = \tau_0 + \eta_p \dot{\gamma} \quad (12)$$

Herschel-Bulkley

$$\tau = \tau_0 + K(\dot{\gamma})^n \quad (13)$$

Newton

$$\tau = \eta \dot{\gamma} \quad (14)$$

Oswald de Waele

$$\tau = K(\dot{\gamma})^n \quad (15)$$

In equations (12)-(15): τ represents the shear stress (Pa), $\dot{\gamma}$ represents the shear rate (s^{-1}), η represents the viscosity of the samples (Pas), τ_0 represents the yield stress (Pa), η_p represents Bingham's plastic viscosity K , represents the consistency index ($Pa \cdot s^n$), and n represents the flow behaviour index (dimensionless).

The samples' flow behaviour index (n) categorises the flow into either Newtonian or non-Newtonian flow while consistency index represents the ease of the sample's flow when sheared (RheoSense Inc, 2015). Newtonian flow was represented by $n=1$ and in contrast, non-Newtonian flow is represented by $1 < n > 1$. The application of the Oswald de Waele flow model was to illustrate the relationship between the viscosity and the shear rate. Bingham flow model was used to determine the yield stress necessary to start the flow by overcoming the viscous resistance of the samples. Therefore, fitting results into the models estimates the behavioural variations of the samples' parameters at the unmeasured shear rate.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Physicochemical Characteristics of Oils

4.1.1 Acid Value and Free Fatty Acid

Acid values of the selected feedstock oils are as depicted in Fig. 4. It was observed that CNSL had high levels of acidity. Elevated levels of acidity in the sample was due to the presence of a considerable amount of moisture in the sample 5.53%, as presented Fig. 10 (Udoh *et al.*, 2017). Furthermore, a study by Kyei *et al.* (2019) reported high acid value in CNSL 12.10-15.40 %, and the study stated that the acidity of the extracted liquid was impacted by the extraction method utilized for obtaining the liquid. The impact of high acid values of the feedstock oil in biodiesel production is that conversion efficiency is lowered since saponification reaction occurs hence producing less biodiesel.

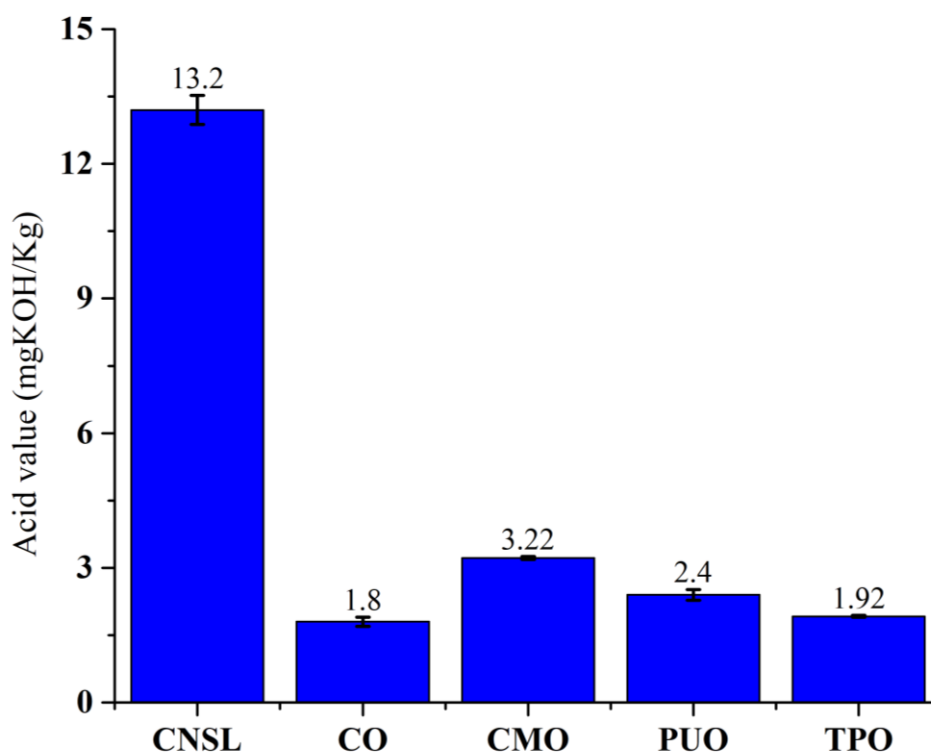


Figure 4: Acid values of the feedstock oils

The FFA profile of the feedstock oils is shown in Fig. 5. Free Fatty Acid profile determines the path taken in the biodiesel production process. The results showed the FFA profile of CO, CMO, PUO, and TPO is $\leq 2\%$, suggesting that biodiesel synthesis from these oils can be

achieved by a single-stage process (transesterification process) (Bouaid *et al.*, 2016; Suraj *et al.*, 2020). The low composition of FFA in the samples showed that the sample oils were still fresh and of high quality. Conversely, the CNSL FFA profile is >2%, suggesting that the route biodiesel manufacturing process could be achieved through a double-stage process that requires excess alkali to neutralise the oil's acidity and be used as a catalyst as well (esterification followed by transesterification process). High FFA composition causes the conversion of the oils into soap since there are many carboxylic groups in the sample. Therefore, to inhibit soap formation reactions and stimulate the transesterification reaction some of the FFAs are to be neutralized by using a strong alkali, which will lead to the increase in biodiesel yield (Bouaid *et al.*, 2016).

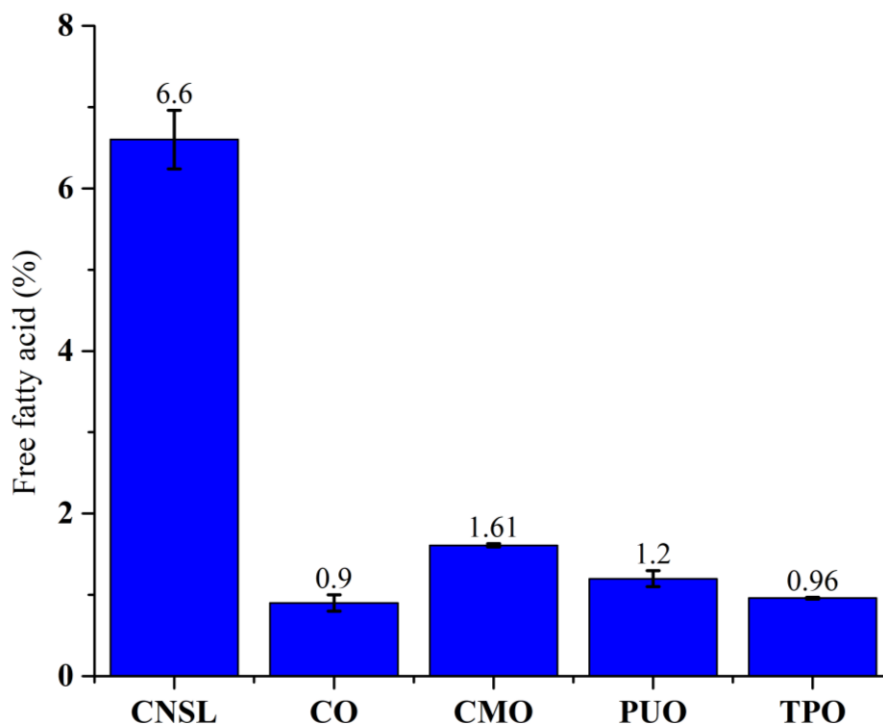


Figure 5: Free Fatty Acids profile of the feedstock oils

Since FFA is a derivative of the acid value, high FFA composition in CNSL was as well a result of the high moisture content in the sample oil (Udoh *et al.*, 2017). Excess moisture in the oil caused a hydrolytic rancidity, the breakdown of fats into acids. The results revealed that the FFA composition of the selected vegetable oils correlated with other studies conducted in East Africa. A study by Kivevele and Mbarawa (2010), and Uwiragiye and Anyiam (2020) stated that the FFA profile of CMO is 1.73% and 1.54%, respectively. Omari *et al.* (2015) reported that the FFA profiles in CO from different regions of Tanzania varied from 0.22% to 0.99%.

4.1.2 Saponification Value

Saponification values ranged from 66.8 mg KOH/g to 190.7 mgKOH/g as shown in Fig. 6. The saponification values of CMO and PUO were relatively similar, 190.7 mg KOH/g and 190 mg KOH/g, respectively. The resemblance of saponification values suggests that incorporated oil samples of CMO and PUO in this study were composed of carbon-carbon bonds with the same molecular weight and carried similar functional groups (Toscano *et al.*, 2012; Uwiragiye & Anyiam, 2020). The similarity in saponification values resulted from the direct linkage between the saponification value and the molecular weight of the triglyceride chains (fatty acid chains) present in the oil. While all the other sample oils possessed saponification values that agree with the commended saponification values reported in the literature by Chavan *et al.* (2018), Kyei *et al.* (2019) and Omari *et al.* (2015) and the observed value for PUO was slightly higher compared to the literature by Minzangi *et al.* (2011). The literature reported the saponification value of PUO extracted from the seeds collected in Kivu, DRC was 182.5 mg KOH/g and the deviation could be attributed to the differences of ecological habitat features such as the soil's primary nutrients, climatic conditions and the altitude of the area the plant was grown (Chuah *et al.*, 2016; Minzangi *et al.*, 2011). The mentioned factors influence the plant's nutrient uptake causing minor variations in the properties of the oils. Also, the difference was possibly because of the chemical composition of impurities that were present in the sample oil (Uwiragiye & Anyiam, 2020). However, not enough amount of research based on PUO has been conducted.

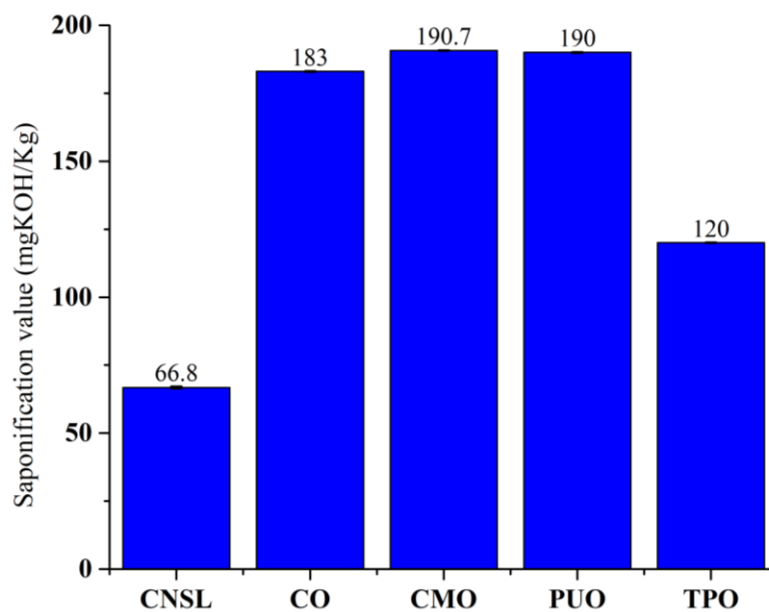


Figure 6: Saponification values of the feedstock oil

4.1.3 Peroxide Value

Presented in Fig. 7, except for CNSL, peroxide values of oils observed were <10 meq/kg, verifying that the samples used were still fresh. The peroxide value results of CO, CMO, PUO and TPO suggest that the method used for extraction of the oil prevented oxidation from occurring hence the quality of the feedstock was maintained. Furthermore, it showed that the samples had a longer shelf life which means it would be some time prior to the sample's deterioration. The peroxide value of CNSL was 43.1 meq/Kg, relatively higher, which could be attributed to the oxidation reaction occurring during the oil extraction process, which involved roasting the shells (Kyei *et al.*, 2019; Rodrigues *et al.*, 2011). Also, high moisture content contributed to the oxidation of the liquid and resulted in a high peroxide value. The peroxide value of CO was similar to that reported by Omari *et al.* (2015) which was 10.79-13.73 meq/kg. For CMO the reported peroxide values were 4.87-8.66 meq/Kg (Kumar *et al.*, 2016). Therefore, the peroxide values of all other oil samples were in synchronization with those reported in other literatures.

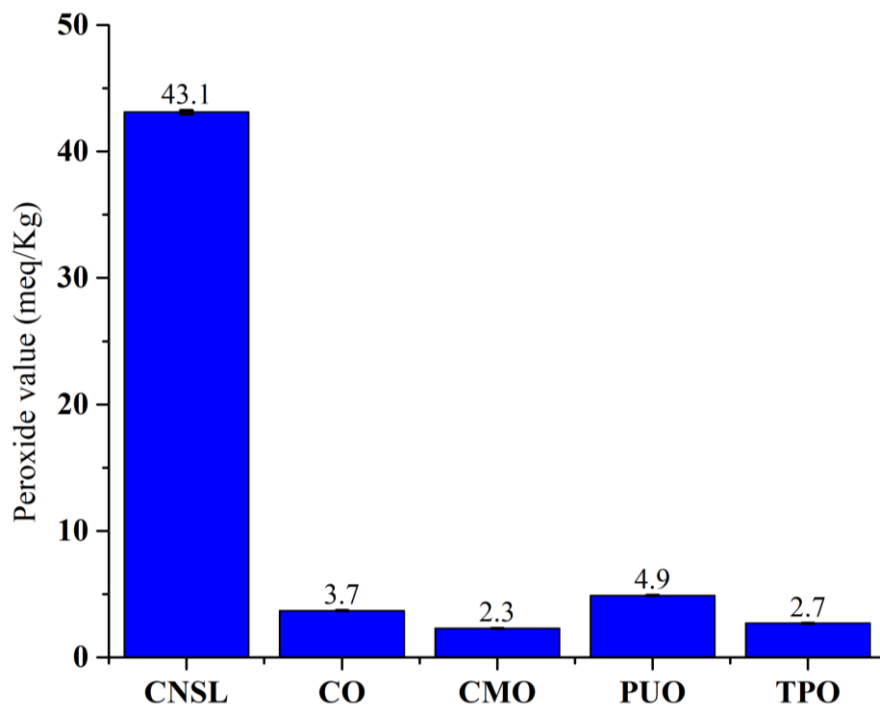


Figure 7: Peroxide values of the selected oils

4.1.4 Iodine Value

As reported in Fig. 8, the iodine values recorded in this study were between 82.8 mgI₂/100 g to 126.5 mgI₂/100 g. It was observed that CNSL and CMO had the highest iodine values 124.4

mgI₂/100 g and 126.5 mgI₂/100 g respectively. The values suggested that the oil samples possessed a considerable amount of unsaturated fatty acids and had low oxidative stability characteristics (Shaah *et al.*, 2021). A study by Shalaby (2015) stated that non-edible oils were characterised by high iodine values and hence have low oxidative stability. Also, the iodine values of CNSL and CMO were comparatively similar, which could be accredited to an approximately equal number of unsaturated carbon-carbon bonds in the fatty acids. However, the value of CMO observed in this study was less than that observed by Kumar *et al.* (2016), which was 139.2 mgI₂/100 g. Similarly, the observed iodine value of CNSL was lower than the reported standard value of 250 mgI₂/100g. The difference could be the account of thermo-oxidative alteration during the extraction process that caused the unsaturated carbon bonds of fatty acids to break (Patterson, 2011; Suraj *et al.*, 2020). Furthermore, high degree of unsaturation of the CNSL and CMO suggests that the feedstock oils are to instantly be used for biodiesel's manufacturing process so that the feedstock's quality is not compromised. However, the biodiesel produced is also most likely to have unsaturation degree of less than 3-8 hours per ASTM6751 07b and EN14214 standards recommendations. To prevent degradation from occurring CNSL and CMO feedstock is to not be exposed to the atmosphere or stored in facilities that are designed with active metals including but not limited to aluminium (Al) and iron (Fe). Designs with unreactive materials as silver (Ag) or designs of active materials coated with unreactive materials are appropriate.

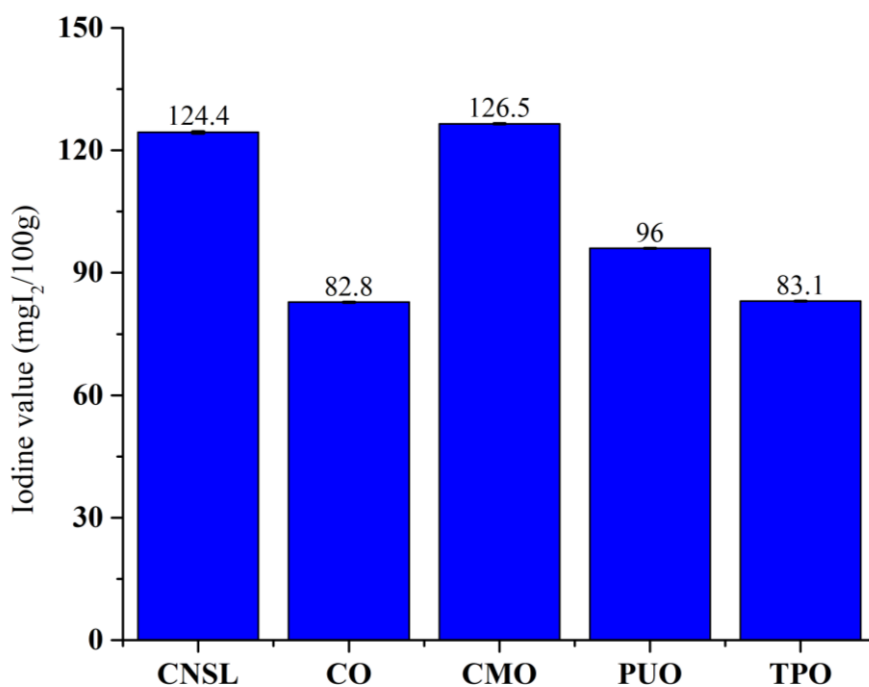


Figure 8: Iodine value representing the extent of unsaturation in the feedstock oil

4.1.5 Specific Gravity

As reported in Fig. 9, the specific gravity values of oils were between 800 and 960. Specific gravity values of CNSL and CO samples were within the set standard range, which is 950–970 and 968–975.7 for the feedstock oils, respectively (Eke *et al.*, 2019; Omari *et al.*, 2015). Average densities of the sample ensure occurrence of proper mixing of the raw materials during the production process. Also, it indicates the agitation speed necessary to mix the raw materials and speed up the rate of the reaction is to be average. Furthermore, it ensures the produced biodiesel will be of quality with proper and ease fuel exhaustion. Moreover, since the oils' specific gravity is average, the use of high agitation speed during the production process could result to over-processing of reactants forming a new reaction between the produced biodiesel (methyl esters) and the catalyst (alkali) resulting to soap formation. Soap formation degrades the quality and conversion efficiency of the produced biodiesel.

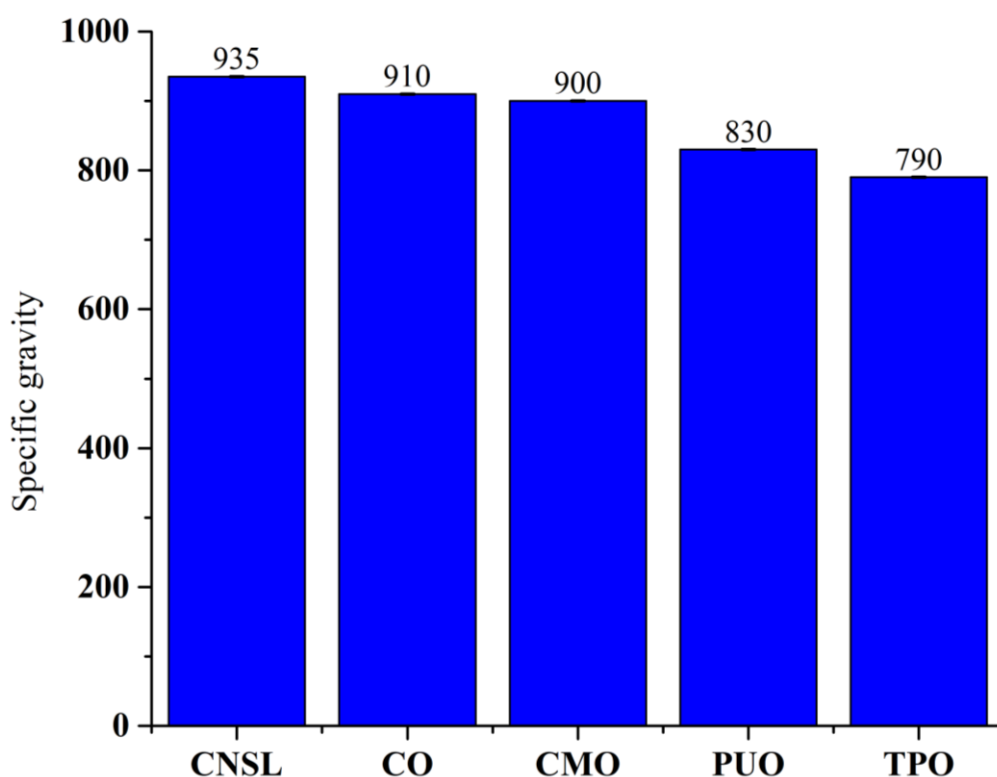


Figure 9: Specific gravity of the feedstock oils

4.1.6 Moisture Content

Moisture content in CO, PUO, and TPO was within the accepted standard range recommended by ASTM6751 07b and EN14214 which is $\leq 0.05\%$ for maintenance of the oil quality (Iqbal *et al.*, 2011; Suwari *et al.*, 2017). However, in Fig. 10, the moisture content values in CNSL and CMO were 5.53% and 1.1% respectively. Moisture content in CNSL was extremely high with respect to recommended standard value and could be attributed by the method used in the extraction process. The extraction process of CNSL involved roasting the shells in the presence of water which prevents combustion from occurring (Kyei *et al.*, 2019). High moisture content in CNSL increased the rate of degradation of the oil (Kyei *et al.*, 2019). As for CMO, high moisture content might be a result of the presence of pre-mature seeds which possessed high moisture content compared to the oil content (Aga *et al.*, 2020). The studies show that moisture content in biodiesel feedstock compromised the quality of the feedstock. Also, it affected the production efficiency of the oil because of soap formation during the process (Ana Godson & Bassey, 2015; Shaah *et al.*, 2021).

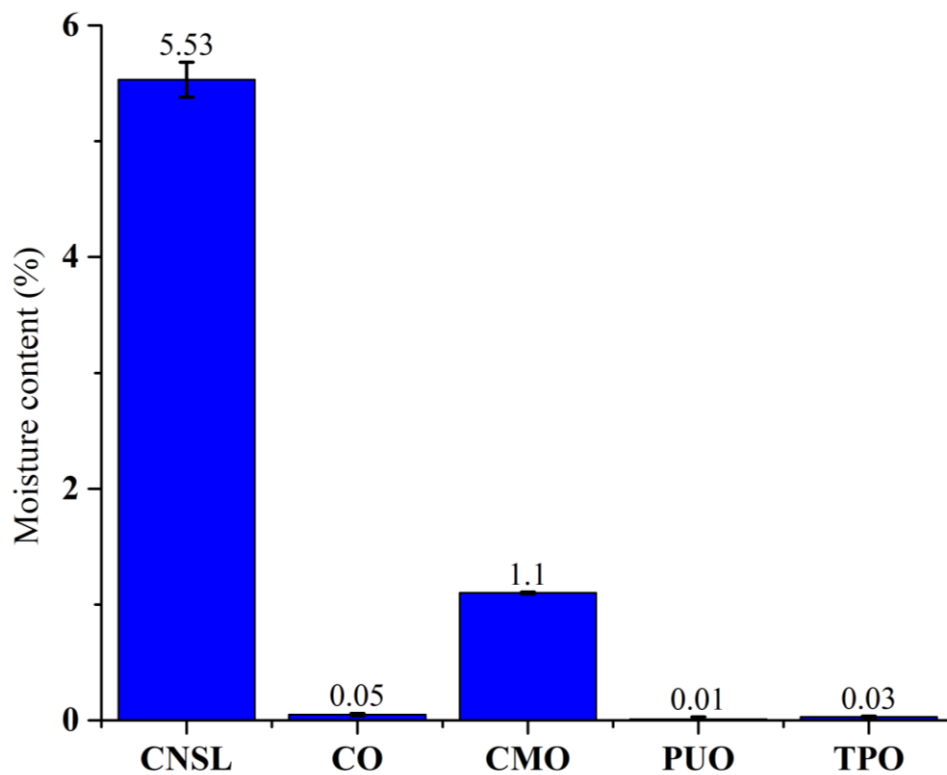


Figure 10: Moisture content profile of the feedstock oils

To resolve the drawbacks caused by the high moisture content of the feedstock, it is recommended for the oils be dried at low temperatures to moisture content of $\leq 0.05\%$.

4.1.7 Oil Yield

After extraction, the yield of vegetable oils was within the range of 29-65%. *Croton megalocarpus* seeds had the lowest yield of only 29%, below the average range of 30%-32% reported by Aliyu *et al.* (2010) and Wu *et al.* (2013). However as shown in Fig. 11, the yield was above the satisfactory range for biodiesel feedstock, which was at least 20% oil yield from the seeds (Karmakar & Halder, 2019; Zulqarnain *et al.*, 2021). Hence, the selected non-edible oils in this research are all economically suitable to be used for large-scale biodiesel production. Presence of moisture in feedstock oils presents a threat to the feedstock's quality because it emphasizes occurrence of hydrolysis rancidity.

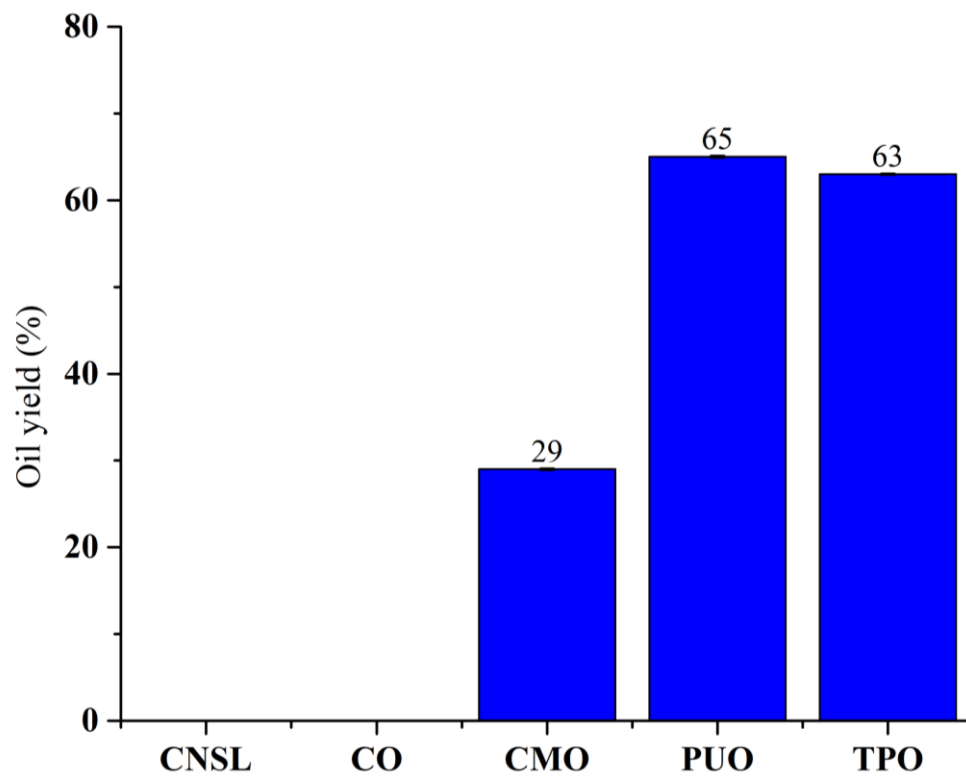


Figure 11: Oil yield of CMO, PUO and TPO

4.2 Rheological Characterisation of The Selected Oils

4.2.1 The Variation of Shear Stress and Dynamic Viscosity with Shear Rate

The internal resistance to flow offered by the selected raw materials that are used in the manufacturing process of biodiesel predetermines the flow behaviour that is to be exhibited by the produced biodiesel (Paul *et al.*, 2021). Therefore, carrying out a thermo-physical analysis of the feedstock in advance determines the suitable production processes and facilities designs

(Silva *et al.*, 2015). Figure 12 presents the computed flow curves that show the shear rate-shear stress relationship of each oil sample at 30°C, 40°C, 50°C, and 60°C. Shown in Fig. 12, for all the samples analysed, a uniform linear correlation of shear stress as a function of shear rate is revealed. For CNSL, CO, CMO, PUO, and TPO, the shear stress shear rate flow relationship observed in this study was that of an ideal liquid that exhibits Newtonian flow behaviour. The results revealed that at zero shear rate, there was no minimum shear stress (y-intercept = 0 Pa) required to break the interactive bonds among the particles of the sample to start the flow of the sample oils. The shear rate applied to the samples was directly proportional to the shear stress exhibited by the samples for overcoming the viscous resistance offered. For each of the samples analysed, the viscosity remained constant regardless of the increase in the shear rate applied implying that the sample oils possessed a Newtonian flow behaviour. To further confirm the observed nature of the flow exhibited by the samples in the study, a linear fit on a power model was carried out to determine the behaviour index of the samples.

$$\ln \tau = n \ln \gamma + \ln K \quad (16)$$

Equation 16 represents Power-law as the equation of a straight line. Flow behaviour index (n) represents the slope of the flow curves, and K is the consistency index representing the y-intercept.

According to the Power model in Tables 3, 4, 5 and 6, the flow behaviour index (n) of the linear fit was carried out for all the samples $0.9877 \leq n \leq 1.1000$. For all the computed values of n , there was an insignificant variation from the actual value of n for substances that exhibit Newtonian flow, which is $n=1$. Moreover, upon fitting the computed data in the respective model, the calculated correlation coefficient R^2 values for all the samples were greater than 0.99. This verified that the linearity of the flow curves and Newtonian flow behaviour exhibited by the sample oils of the samples were appropriately represented by the power law.

A study by Wang *et al.* (2018) which studied rheological characteristics of CO observed similar behaviour. However, no rheological studies have been conducted for other selected vegetable oils used in this research. High values of shear stress were observed during the analysis of CNSL and CO samples because of the oil's high viscous resistance. The high viscosity of these samples is associated with strong intermolecular cohesive forces that resist the motion of the molecules. As a result, high torque is required to overcome the intermolecular force resistance for the flow to occur. The average viscosity of CNSL, CO and TPO observed in this study

ranged between 0.047-0.300 Pas which is at least two times higher than the average viscosity of edible oils which is 0.0196-0.0217 Pas reported by Silva *et al.* (2015). The average viscosities of CMO and PUO were not significantly different at $p \leq 0.05$ to the average viscosity of canola oil, which was observed to have the highest viscosity among the other edible oils assessed in the study. Sahasrabudhe *et al.* (2017), reported the average viscosity of canola oil to be lower than that of CMO and PUO. High viscosity of the assessed oils in this study results from high FFA composition in non-edible oils compared to edible oils (Bouaid *et al.*, 2016).

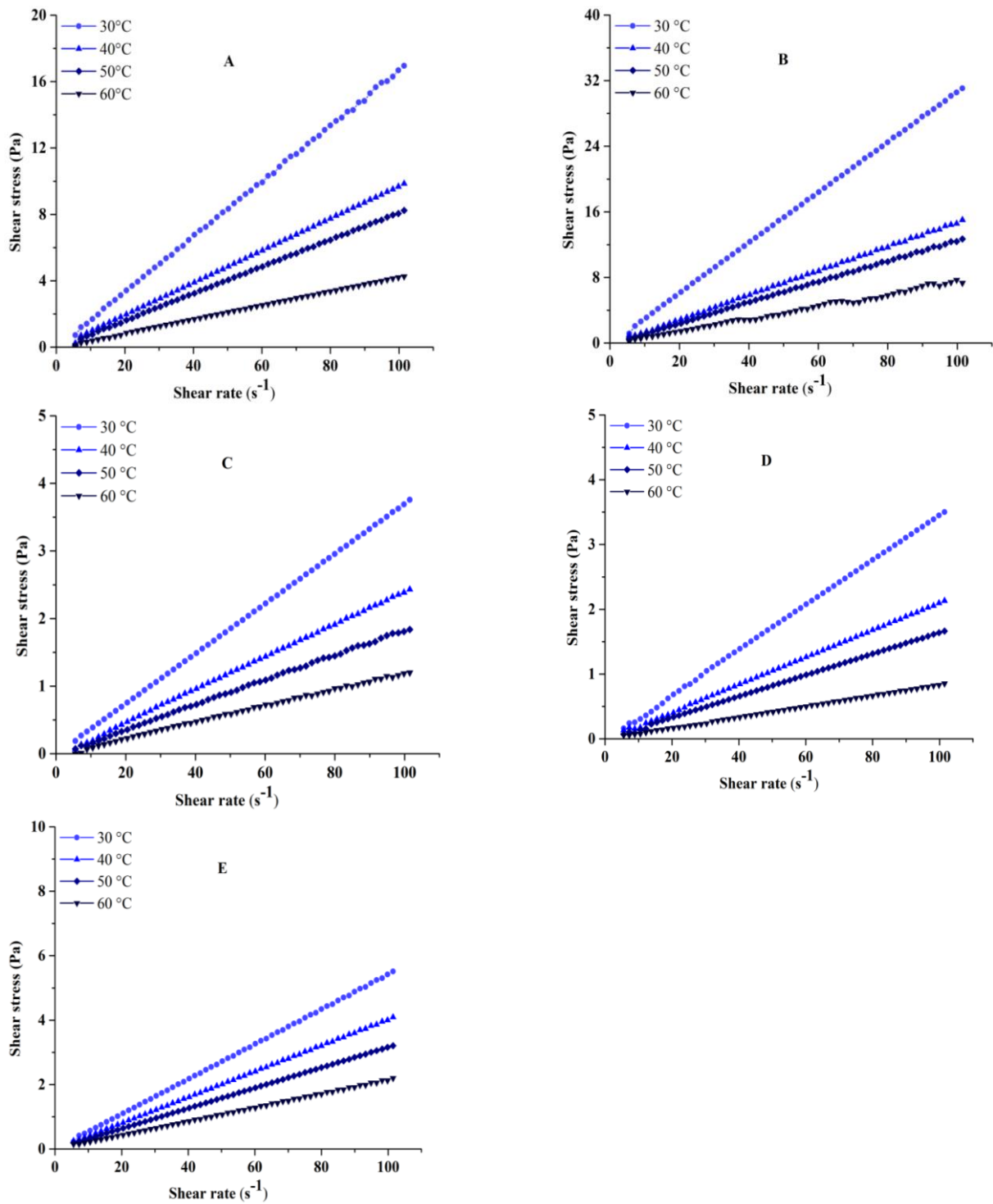


Figure 12: Flow curves of sample oils at the temperatures 30 °C, 40 °C, 50 °C and 60 °C. (A) Cashew Nut Shell Liquid, (B) Castor Oil, (C) Croton *Megalocarpus* Oil, (D) Podocarpus *Usambarensis*, and (E) *Thevetia Peruviana* Oil

4.2.2 The Influence of Temperature on Shear Stress and Viscosity

The influence of temperature on shear stress as a function of shear rate was analysed at the temperature of 30°C, 40°C, 50°C, and 60°C. The results in Fig. 13 revealed that, with an increase in temperature shear stress values exhibited by the samples decreased. The shear stress values of CNSL, CO, CMO, PUO, and TPO at the temperature of 30°C and shear rate 100 s⁻¹ were 16.95 Pa, 31.06 Pa, 3.76 Pa, 2.13 Pa, and 5.42 Pa, respectively. At 60°C, the maximum shear stress values observed for CNSL, CO, CMO, PUO, and TPO were 4.25 Pa, 7.32 Pa, 1.20 Pa, 0.52 Pa, and 2.13 Pa, respectively. At the shear rate of 100s⁻¹, the decrease in the shear stress from 30°C to 60°C was 74.5% for CNSL and CO, 31.9% for CMO, 24.4% for PUO, and 39.3% for TPO. Also, the viscosity of all the samples analysed decreased as the temperature increased. The decrease in viscosity resulted in the low torque required to overcome the viscous resistance offered by the sample (Wang *et al.*, 2018). This describes the decrease in shear stress, which resulted from the decrease of dynamic viscosity in the samples as the temperature increased, similar results were reported by Sahasrabudhe *et al.* (2017) and Wang *et al.* (2018). Figure 13 shows the effect of temperature on the dynamic viscosity of the samples. Drastic alterations of dynamic viscosity in CNSL and CO samples were observed as the temperature increased because the samples had carbon-carbon chains with a high unsaturation degree of FFA (Paul *et al.*, 2021). Unsaturated bonds were deformed under heat (temperature), bringing about drastic changes in the viscosity of the samples. Conversely, the viscosity of CMO, PUO, and TPO steadily decreased, which implied that FFA bonds in the oil were not substantially deformed at the temperature increment. The results show that, regardless of the temperature variations, all samples maintained constant linear flow behaviour, with constant viscosity resulting in the flow behaviour index of approximately equal to 1 at 30°C, 40°C, 50°C, and 60°C.

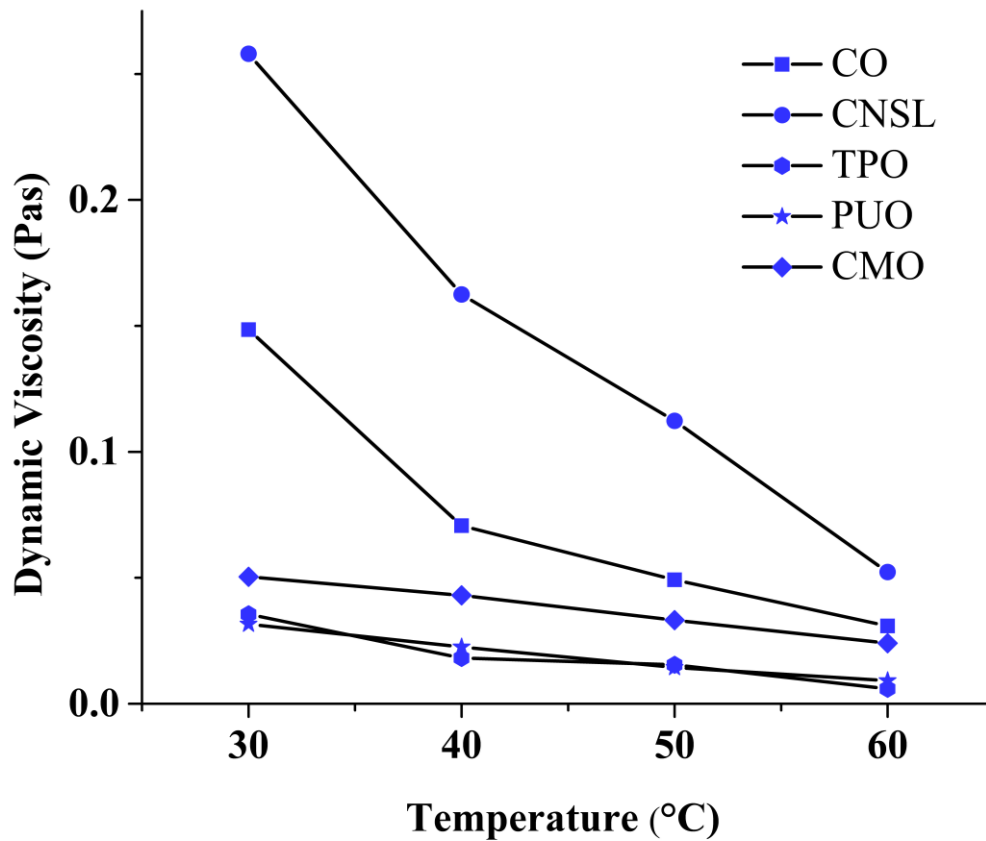


Figure 13: The variation of dynamic viscosity (Pas) with the increase in temperature

However, as shown in Fig. 14, the viscosity of PUO was comparatively similar to that of CMO. Also, as the temperature varied, the dynamic viscosity values comparison was still maintained. The Tukey test confirmed the resemblance in mean dynamic viscosity values at $p \leq 0.05$, as shown in Fig. 14. The results suggested the samples had equal internal resistance to flow, which could be contributed by the resemblance of the morphology of CMO and PUO (Allouche *et al.*, 2003; Song *et al.*, 2021). The similarities in morphology could include but are not limited to the inner structure of the particles, shape of the particles, arrangement of the particles, and size of the particles in the samples. Thus, the torque required to start the flow in CMO was equivalent to that of PUO. As a result, there was no significant difference in the shear stress exhibited at the respective shear rate for both CMO and PUO.

Since the viscosity of the samples was maintained at uniform temperatures, it implies the molecular structure and the molecular arrangement of the molecules were as well maintained throughout the experiments. A study by Buchanan (2017) reported that Newtonian fluids possess isotropic molecules which have a symmetrical shape. These molecules are not

oriented by the shear rate applied in the samples, therefore; not causing any changes in the flow properties of the oils. If the molecules are oriented by the force applied into the sample, dilatant or pseudoplastic flow of the samples would be observed. Molecules orientation changes the direction cause of the molecular flow resulting to the deviation of the direction of the shear stress.

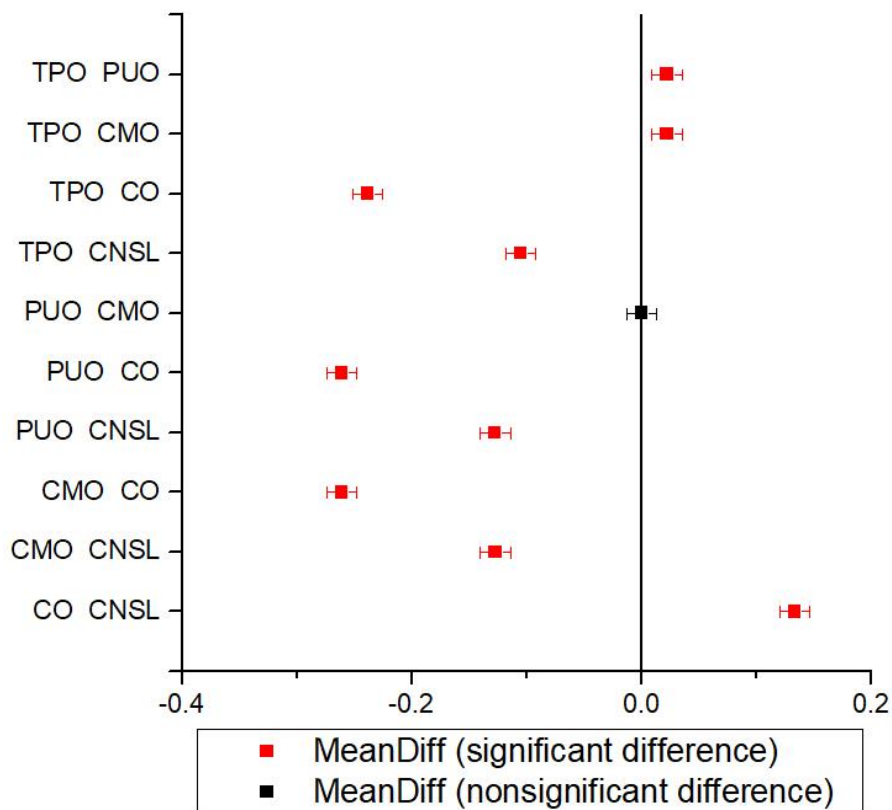


Figure 14: Dynamic viscosity means comparison using Tukey's test

Also, according to Power-law, the consistency index K , which describes the ease of flow for each sample, decreased as dynamic viscosity decreased. The computed data revealed that the consistency index fluctuated with the temperature increase, which decreased with an increase in temperature. Low consistency index values were observed at high temperatures, which suggested that the samples flow readily at high temperatures and vice versa. The ease of flow at high temperatures results from the weakened intermolecular chains that hold up the carbon-carbon bonds together (Hirschfelder, 2009; Song *et al.*, 2021). The computed rheological parameters for each model selected and for all the samples at 30°C, 40°C, 50°C, and 60°C are presented in Tables 3, 4, 5, and 6.

Table 3: Rheological models parameters at 30°C

At 30 °C												
Rheological model	Newton		Bingham			Oswald de Waele			Herschel Bulkley			
	$\tau = \eta \dot{\gamma}$		$\tau = \tau_0 + \eta_p \dot{\gamma}$			$\tau = K(\dot{\gamma})^n$			$\tau = \tau_0 + K(\dot{\gamma})^n$			
Oils	η	R^2	τ_0	η_p	R^2	K	n	R^2	τ_0	K	n	R^2
CNSL	0.1604	0.999	-0.0646	0.1617	0.999	0.1729	0.980	0.999	-0.5611	0.2844	0.8741	0.996
CO	0.2949	0.999	-0.3427	0.2995	0.998	0.2769	1.014	0.999	-0.6765	0.3557	0.9643	1.000
CMO	0.0334	1.000	0.0000	0.0334	1.000	0.0335	1.000	1.000	-0.0001	0.0335	1.000	0.999
PUO	0.0340	0.992	-0.6760	0.0357	0.993	0.02219	1.070	0.993	-0.7259	0.0374	0.9927	0.993
TPO	0.0565	0.990	0.1322	0.0548	0.991	0.09427	0.996	0.993	-0.3703	0.1682	0.9985	0.994

Table 4: Rheological models parameters at 40°C

At 40 °C												
Rheological model	Newton		Bingham			Oswald de Waele			Herschel Bulkley			
	$\tau = \eta \dot{\gamma}$		$\tau = \tau_0 + \eta_p \dot{\gamma}$			$\tau = K(\dot{\gamma})^n$			$\tau = \tau_0 + K(\dot{\gamma})^n$			
Oils	η	R^2	τ_0	η_p	R^2	K	n	R^2	τ_0	K	n	R^2
CNSL	0.09410	0.995	0.04774	0.0935	0.959	0.1381	0.991	0.997	-0.9445	0.3314	0.7412	0.998
CO	0.1336	0.999	-0.0652	0.1345	0.999	0.1234	1.018	1.000	0.0180	0.1215	1.021	0.978
CMO	0.01769	0.997	0.02865	0.0173	0.998	0.02508	0.9980	0.999	-0.0619	0.3533	0.8530	0.995
PUO	0.01749	1.000	-0.0005	0.0175	0.993	0.01747	1.000	1.000	-0.0012	0.0176	0.9985	0.993
TPO	0.03732	1.000	0.00956	0.0372	0.999	0.03937	0.9878	1.000	-0.0134	0.0410	0.9798	0.999

Table 5: Rheological models parameters at 50°C

At 50 °C												
Rheological model	Newton		Bingham			Oswald de Waele			Herschel Bulkley			
	$\tau = \eta \dot{\gamma}$		$\tau = \tau_0 + \eta_p \dot{\gamma}$			$\tau = K(\dot{\gamma})^n$			$\tau = \tau_0 + K(\dot{\gamma})^n$			
Oils	η	R^2	τ_0	η_p	R^2	K	n	R^2	τ_0	K	n	R^2
CNSL	0.0733	0.999	-0.0348	0.0738	0.999	0.07218	1.004	0.999	-0.6423	0.1409	0.8759	1.000
CO	0.1101	0.999	-0.1040	0.1115	0.999	0.09724	1.028	1.000	-0.0378	0.1011	1.020	1.000
CMO	0.0236	0.987	-0.4059	0.0180	0.971	0.1003	0.999	0.990	-0.064	0.0034	1.086	0.929
PUO	0.0185	1.000	-0.0004	0.0185	1.000	0.01861	0.9999	1.000	-0.0004	0.0187	0.9985	0.934
TPO	0.0337	0.998	-0.0378	0.0344	0.983	0.02279	1.096	0.999	0.0728	0.0152	1.186	0.948

Table 6: Rheological models parameters at 60°C

At 60 °C												
Rheological model	Newton		Bingham			Oswald de Waele			Herschel Bulkley			
	$\tau = \eta \dot{\gamma}$		$\tau = \tau_0 + \eta_p \dot{\gamma}$			$\tau = K(\dot{\gamma})^n$			$\tau = \tau_0 + K(\dot{\gamma})^n$			
Oils	η	R^2	τ_0	η_p	R^2	K	n	R^2	τ_0	K	n	R^2
CNSL	0.0355	0.997	-0.1141	0.0335	0.997	0.04695	0.932	1.000	0.0271	0.0435	0.9466	0.946
CO	0.0705	0.995	-0.3354	0.0750	0.997	0.03325	1.100	0.998	-0.0228	0.0347	1.146	0.943
CMO	0.0130	0.997	-0.0534	0.0137	0.981	0.01049	1.049	0.998	-0.1118	0.0248	0.8775	0.999
PUO	0.0150	1.000	-0.0150	0.0002	0.999	0.01520	0.9973	1.000	-0.0030	0.0156	0.9925	0.934
TPO	0.0208	1.000	0.00164	0.0208	0.956	0.02130	0.9950	1.000	-0.0045	0.0218	0.9899	1.000

4.2.3 Determination of the Appropriate Rheological Predictive Model for the Oils

Rheological models that described the flow behaviour of the selected non-edible vegetable oils were assessed by fitting the experimental data into the selected four rheological models. The models were Newton, Bingham, Oswald de Waele (power-law), and Herschel Bulkley's rheological models. The selection of the models was on account of the time-independent behaviour that was portrayed by the rheograms of the samples. The predictive models represented the shear stress-shear rate relationship by employing the introduction of flow predictive parameters in each model. The predictive flow parameters that were used for modelling are described in Tables 3, 4, 5, and 6.

In Newton's rheological model, the determinant predictive parameter was the apparent viscosity η (Pas). For all the samples, it was observed that the apparent viscosity was decreasing with the increase in temperature. As a result, it affected the study's dependent variable, which was shear stress. The correlation coefficient calculated by the experimental data fitting into this rheological model was $R^2 \geq 0.987$. In Bingham's rheological model, the predictive parameters introduced in the shear rate–shear stress relationship were yield stress τ_0 (Pa) and Bingham's plastic viscosity η_p (Pas). Bingham's rheological model focused on the apparent viscosity of the samples at low shear rates that are approximately 0 s^{-1} . The yield stress values exhibited by all the selected samples were $\tau_0 < 0$, which were not in correlation to the accepted values for the Bingham's fluids, which require yield stress that is greater than 0 ($\tau_0 > 0$) to cause flow in the samples at the shear rate of 0 s^{-1} . The Bingham's plastic values for the samples were less than 0.5 Pas, resulting in negative yield stress values calculated. A study conducted by Silva *et al.* (2015) confirmed that edible oils are as well not well represented by Bingham's rheological model implying both edible and non-edible oils require no stress applied to initiate flow in the building blocks of the oils' molecules. The correlation fit of the experimental data into this rheological model was $R^2 \geq 0.956$.

In Oswald de Waele's rheological model, the predictive parameters were consistency index K (Pas^n) and flow behaviour index n . Irrespective of the characterisation temperature, $n = 1$ and $K > 0$ for all the samples. The decrease of the consistency index with the increase in temperature revealed that the viscous resistance was weakened, resulting in oil thinning; hence, the pumping ability of the oils was increased. This observation would also be reflected in the manufactured biodiesel, which would mean that quality fuel atomisation would be achieved at high temperatures. The computed predictive parameter values were correlated to the expected

parameter values for the Oswald de Welle model, and a positive correlation was revealed. The experimental data fit in this model showed a correlation of $R^2 \geq 0.990$. The predictive parameters in the Herschel Bulkley flow model were yield stress τ_0 (Pa), consistency index K ($\text{Pa}\cdot\text{s}^n$), and flow behaviour index n. The calculated yield stress values at 0 s^{-1} were $-0.7259 \leq \tau_0 \leq 0.0728$, which showed that the computed values were out of the expected range of $\tau_0 > 0$ for Herschel Bulkley fluids. However, computed values for n and K were within the expected range for Herschel Bulkley fluids, $0 < n < \infty$ and $K > 0$. The correlation of the fit in the Herschel-Bulkley model was $R^2 \geq 0.929$, which is not the most appropriate fit among the selected flow behaviour predictive models.

According to the correlation coefficient (R^2) values computed by fitting the experimental data into the rheological models, Newtonian, Oswald de Waele, and Bingham's rheological models show an appropriate fit to describe the flow behaviour of the samples. However, Herschel Bulkley's model showed a weak correlation to the data. Also, some τ_0 values in Herschel-Bulkley's model were negative, which contrasts with the expected yield stress of Herschel Bulkley's model, $\tau_0 > 0$. Similarly, the computed yield stress values in the Bingham model were negative, meaning that none of the oils required externally applied stress to start flowing. Hence, Bingham's and Herschel Bulkley's rheological models were not appropriate for presenting the flow behaviour of the selected sample oils. The results of this study showed a resemblance to that of Paul *et al.* (2021), a study that analysed the rheological characteristics of CO and methyl ester. Furthermore, the study reported that at low shear rates of $5 - 100\text{ s}^{-1}$, CO and the manufactured methyl ester portrayed a non-Newtonian flow behaviour. However, in this study, non-Newtonian flow behaviour was not portrayed by any of the selected vegetable oil. Also, a study by Abdelraziq and Nierat (2015) showed that CO had depicted Newtonian flow behaviour irrespective of the shear rate and temperature at which the rheometer was operated. Therefore, all sample oils portrayed Newtonian flow behaviour, which was appropriately represented by Newton and Oswald de Waele's rheological models. Research by Sahasrabudhe *et al.* (2017) and Silva *et al.* (2015) described that a similar flow behaviour was exhibited by edible oils where the flow behaviour index maintained was equal to 1 at elevated temperatures from 20°C . The studies concluded that Newton and Oswald de Waele's rheological models were appropriate to present the flow behaviour of the analysed edible oils.

4.3 Heat Stability Index of Non-Edible Vegetable Oils Against Temperature Variations

Figure 15 presents the heat stability indexes in percentage that were observed for non-edible oils feedstock that are suggested to be used in biodiesel production. It was observed that CNSL and CO had the lowest heat indexes 15% and 14% respectively, suggesting that the sample oils absorbed heat energy effortlessly. It also suggests that during the production processes incorporating CO and CNSL as raw materials, elevated temperatures are not to be used since it would result to excess heat absorption degrading the oxidation stability of the samples and the produced biodiesel. High heat absorption can as well result to permanent deformation of the bonds forming the triglyceride chains by formation of polymers (Jaarin & Kamisah, 2012; Siraj *et al.*, 2017).

Conversely, the heat stability indexes of CMO and TPO were similar and the highest heat indexes of 43%. It infers that CMO and TPO have the lowest ability to absorb heat hence, the production process of biodiesel can take place at elevated temperatures without risking the degradation of the feedstock. Even though it was revealed that CMO and TPO can withstand consistent variation of temperature, oxidation of the sample oils can still occur at elevated temperatures. The alterations of the oil's properties are however not revealed instantly. Also, oils with high heat stability indexes have a low degree of unsaturation. In high thermostability feedstock oils or biodiesel produced, the carbon – carbon bonds forming the triglyceride chains are mostly sigma bonds, with a few unsaturated and polyunsaturated bonds (Paul *et al.*, 2021).

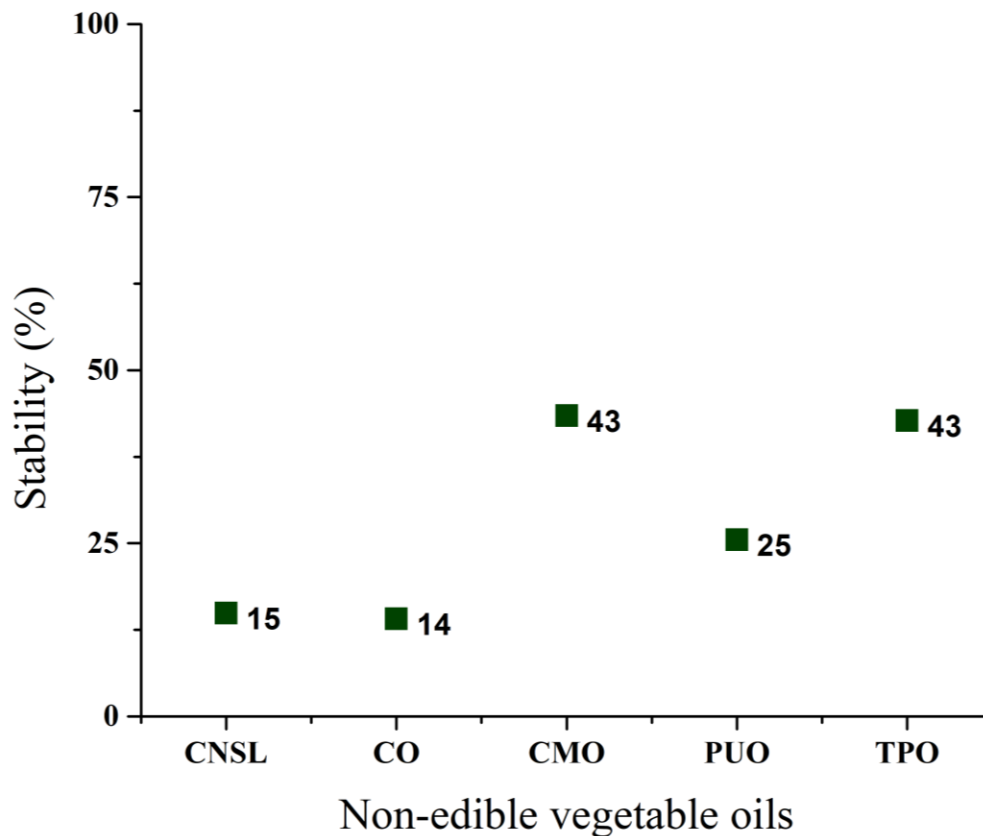


Figure 15: Heat stability indexes of non-edible feedstock oils

Heat stability of the oils describes the resistance of the sample oils' physical properties such as density, viscosity and the colour observed to be altered with the temperature variation. For fuels, heat stability is of significance since it ensures there is an occurrence of a steady fuel atomization and exhaustion (Silva *et al.*, 2015). Stability of the biodiesel feedstock influences a uniform mixing of the reactants at varied temperatures, determines the ease of the produced fuels degradation and suggests the temperatures below which over processing of the reactants can occur (Istiningrum *et al.*, 2017). The use of fixed temperatures for a variety of feedstock during biodiesel production process is not advised since the resistance of the feedstock's ability to absorb heat varies. The feedstock's resistance to changes caused by temperature variations is also described in terms of the saturation degree of the oils.

4.4 Pathway of Biodiesel Production from Non-Edible Oils

Since physicochemical characterization of the non-edible feedstock revealed that CNSL had FFA content of 6.6%, thus pre-treatment of the feedstock is necessary. Depending on the FFA content of the feedstock, the pathway followed during biodiesel's production process is selected so as to increase the conversion efficiency of the feedstock to biodiesel. Figure 16 shows the pathway of the production of biodiesel from high FFA feedstock. The extracted feedstock oil high acid value is neutralized by an acid catalysis reaction called esterification. Esterification occurs upon the reaction of a carboxylic acid (organic acid) with alcohol in the presence of a strong acid. The acid that is mostly used for this reaction is sulphuric acid. The reaction is allowed to take place for 1 hour at 60°C. Esterification reaction converts the FFA molecules into esters that cannot form reaction with an alkaline catalyst in the second stage of production.

Acid catalysis is followed by alkaline catalysis also known as the transesterification reaction in which biodiesel is produced from the pre-treated feedstock. In transesterification reaction, the triglycerides and esters in the feedstock are converted to methyl esters (biodiesel) and glycerol. Transesterification reaction takes place when the feedstock is reacted with alcohol in the presence of a strong alkali catalyst. The most common catalysts used are KOH and NaOH. The produced biodiesel is then washed using water to remove the impurities in the fuel produced. The impurities include the residual alkaline catalyst from incomplete reaction, free glycerol, residual alcohol, water and sediments.

The oils from the seeds of castor, croton *megalocarpus*, podocarpus *usambarensis* and *Thevetia peruviana* had the FFA profile of < 1%, suggesting that no pre-treatment is necessary before being used for biodiesel production though, can as well be carried out. Hence, the oil extracted can directly be used for the transesterification reaction to produce biodiesel. Figure 17 shows the pathway of biodiesel production from low FFA profile feedstock.

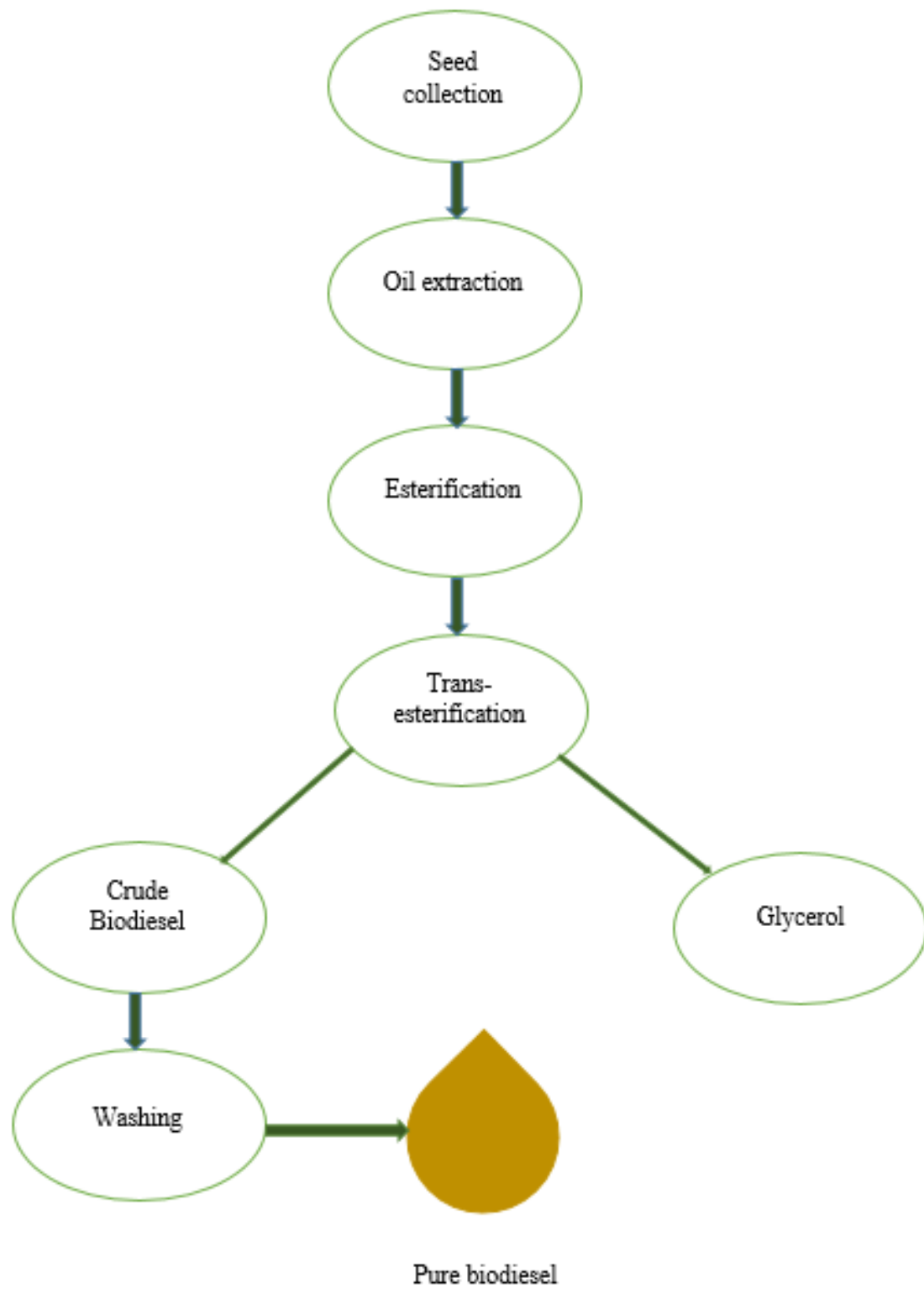


Figure 16: Biodiesel production from CNSL

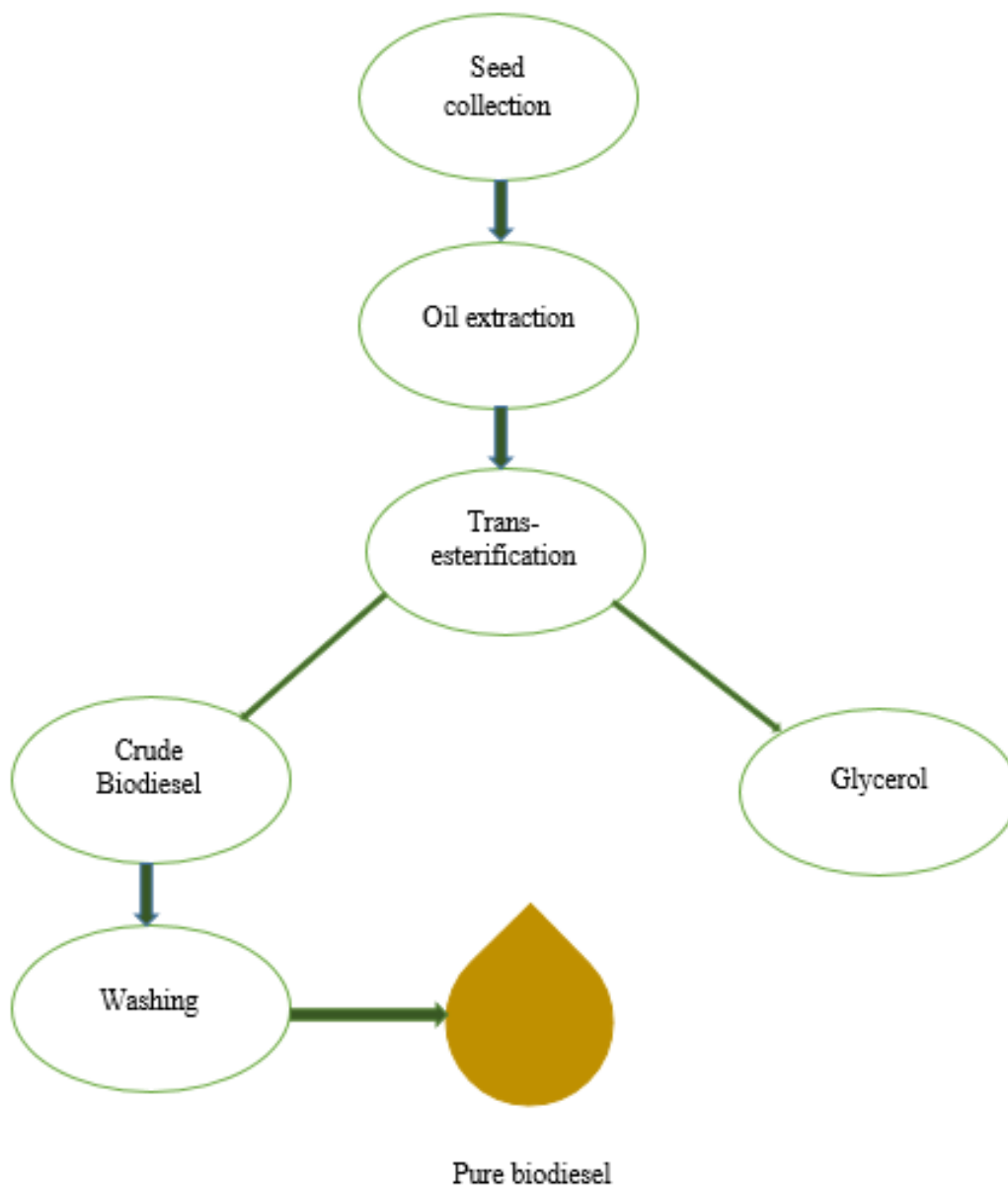


Figure 17: Biodiesel production from CO, CMO, PUO and TPO

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Second generation feedstock being the most affordable biodiesel resource compared to various feedstock types available, it is the future of biodiesel industry. Exploring, studying and utilizing new and existing non-edible feedstock oils for biodiesel manufacturing unlocks the potential of biodiesel replacing mineral diesel by more than 50%. The study focused on characterizing underexploited non-edible oils that can be used to manufacture biodiesel in Africa. Moreover, the study analysed the potential of CNSL, CO, CMO, PUO and TPO to be used for large scale biodiesel production by studying the physical, chemical and flow parameters of the feedstock.

The results conclude, all the selected vegetable oils in this study had an oil yield of 29-65% from the seeds. High oil yield shows that all the studied non-edible vegetable oils are commercially suitable for producing biodiesel at a reasonable cost. However, the results revealed a difference in non-edible physical and chemical properties, solidifying the emphasis on the suitable designs of production facilities. Suitable designs should withstand the high viscous resistance offered by non-edible oils during the mixing process of raw materials. Rheological characterisation revealed that, at a constant temperature, all selected non-edible vegetable oils exhibited Newtonian flow behaviour. All the sample oils used in this study maintained the same behaviour at 30-60°C. Cashew Nut Shell Liquid (CNSL) and CO had high viscosity among all the selected sample oils. In correspondence to their high viscosity nature, the produced biodiesel from CNSL and CO will perform poorly in cold climates. The lowest viscosity of non-edible oils observed in this study is at least twice the viscosity observed in edible oils. High viscosity of CNSL and CO results in the increased cloud point, pour point and flash point, which compromise the quality of fuel atomisation. Though in-tank fuel heaters can resolve this issue, the method consumes enormous energy, which restricts biodiesel's affordability aspect.

The temperature increase caused the decrease in viscosity and consistency index of the samples. The viscosity of CMO and PUO did not show significant difference at $p \leq 0.05$. The rheological characteristics of the selected samples were appropriately represented by Newtonian and Oswald de Waele's rheological models. The rheological models showed a strong correlation to the fit of the experimental data ($R^2 > 0.987$). Therefore, for optimising the biodiesel

manufacturing process from non-edible oils, the average temperature for high viscosity oils such as CNSL and CO is to be maintained at 30-45°C to avoid irreversible deformation of the molecular structure of the produced biodiesel resulting in the fuel's quality compromise. However, for low viscosity non-edible oils, there were no observed effects that could compromise the fuel's quality at elevated temperatures. Correspondingly, the shear rate increase did not show effects on the oil's viscosity, which guaranteed the preservation of the fuel's quality all through the production process. The shear rate was directly proportional to shear stress, which could result to pressure build up in the manufacturing facilities (tanks) in case of high agitation speeds. Lower agitation speed should be maintained to avert the damage to the manufacturing facilities that could result from the exerted pressure from the oils. Thus, new designs of manufacturing systems that can be operated at high shear stress exerted by non-edible oils are recommended.

5.2 Recommendations

- (i) To counteract high viscosity challenge in CNSL and CO feedstock blend may be engaged during biodiesel production. The blend should comprise of high viscous feedstock and low viscous feedstock such as CMO, PUO and TPO. Research is to be conducted to assess the interference of free fatty acids between the oils, its effects to the feedstock's quality and its influence on the produced biodiesel. Additionally, appropriate feedstock mixing ratios are to be determined.
- (ii) Due to the similarity in the properties of CMO and PUO, a blend of the two oils is highly recommended however, research should be conducted to assess the interference of free fatty acids between the oils, its influence on the produced biodiesel and life cycle of the feedstock and the produced biodiesel should be assessed.
- (iii) Also, since it was observed shear stress increased with an increase in shear rate, during the production processes excessive and aggressive mixing of the reactants can result pressure build up in the plant and cause facilities damage. Hence, the appropriate agitation speed is in the range of 400 rpm to 800 rpm (shear rate range $6.7-13.3s^{-1}$), to avoid pressure build up in closed production facilities. Additionally, high agitation speed can result to saponification reaction particularly with non-edible oils being used as the feedstock. Because of high FFA composition in non-edible oils, high agitation

speed will cause the catalyst to react with the produced biodiesel to form soap and stunt conversion efficiency.

- (iv) Economic analysis study focusing on large biodiesel production from non-edible feedstock in Africa highly recommended.

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RESEARCH OUTPUTS

(i) Research Paper

Zakaria, F., Lujaji, F., & Kivevele, T. (2022). Rheological and Physicochemical Analysis of Nonedible Oils Used for Biodiesel Production. *ACS Omega*, 7(42), 37133-37141.

(ii) Poster Presentation

Poster Presentation



RHEOLOGICAL AND PHYSICOCHEMICAL ANALYSIS OF NON-EDIBLE OILS USED FOR BIODIESEL PRODUCTION

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Introduction

Rheological and physicochemical characteristics of biodiesel feedstock are used for quality control in production, storage and transportation processes. This study was focused on establishing rheological and physicochemical characteristics of non-edible oils selected that are cashew nut shell liquid (CNSL), castor oil (CO), croton *megalocarpus* oil (CMO), podocarpus *usambarensis* oil (PUO) and *Thevetia peruviana* oil (TPO).

Materials and Methods

Association of Official Analytical Chemists (AOAC) International standard analysis methods which are titrimetry and gravimetry based methods were used for establishing physicochemical characteristics. Rheological characteristics were studied using shear rheometer, Haake Viscotester, model VT 550, Karlsruhe, Germany.



Results and discussion

Physicochemical characteristics that the studied non-edible oils were all suitable for biodiesel production. Also, all oils exhibited Newtonian flow behaviour.

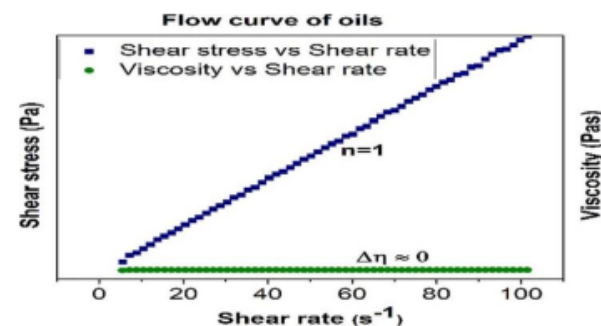


Figure 2: Representation of the flow curves exhibited by the oils

Conclusion

Non-edible oils studied were all suitable for large scale production and during the production process average agitation speeds are to be observed to avoid damage to the production facilities.