



Bioethanol, Neem Oil Methyl Ester and Diesel Blends as Fuel for Diesel Engines: Production and Characterization

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ABSTRACT

*In this research, biodiesel was produced from neem oil using the transesterification method. Bioethanol was produced from cassava peels using *Aspergillus niger* and *Saccharomyces cerevisiae* by simultaneous saccharification and fermentation. The bioethanol-biodiesel blends (BD5BE5D90, BD20BE10D70 and BD40E30D30) were characterised and evaluated for oxidative degradation during 21 days of storage. The oxidative degradation was investigated by UV-Vis spectroscopy and Fourier Transform Infrared Spectroscopy (FTIR). In addition, this study determined the properties of the blends, such as relative density, kinematic viscosity and flashpoint. However, the results from the experiments performed, showed that BD5BE5D90 and BD20BE10D70 blends have relatively low flash points, thereby having the potentiality of ignition in high temperatures and can easily degrade over time, while BD40BE30D40 blend properties are within the EN standards. BD40BE30D40 blend showed significantly better results when compared with the other blends, and there was no oxidative degradation during the storage period.*

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INTRODUCTION

The demand for alternative fuel sources that will replace the existing fossil fuel sources (premium motor spirit and diesel) for the automobile industry is increasing - as people continue to seek cleaner energy sources (Azeez et al., 2019). However, biodiesel can substitute for gaseous fuels to decrease the emissions released from an automobile. Crude oil, a depletable resource, is believed to be scarce in the near future. Although most engines' fuel economy is greatly improved, the rapid increase of automobiles globally is indicative that the demand for fuel will experience a corresponding increase. Therefore, the quest to seek alternative fuel sources has been on the rise lately. In addition, the huge greenhouse gas emissions associated with the internal combustion engine are another motivation to develop alternative fuels (Merlin et al., 2015; Liu et al., 2019; Deepanraj et al., 2015). Biodiesel is a renewable, sustainable and economical energy source that can significantly enhance engine performance and

reduce harmful gaseous discharge. As a result, it was widely created and used by most energy consuming nations globally (Dwivedi et al., 2019). Generally, fuel is a mixture of fatty acid alkyl esters made from vegetable oils, animal fats or recycled greases. Where available, biodiesel can power the present compression ignition engines without any hardware changes in the existing engine setup, and it significantly reduces the emissions of CO and CO₂ by 50% and 80%, respectively that leading to global warming, which is a serious matter of concern worldwide (Leduc et al., 2009). Few researchers have worked to replace conventional fuels with alternative biofuels. Rajesh et al. (2019) studied optimising biodiesel production from waste frying palm oil using a superior screening design. It was observed from the results that a maximum yield of 96.23% was achieved at a molar ratio of 6:1 and reaction temperature of 55°C, and catalytic concentration of 1 wt%, and the method of definitive screening gave maximum yield. The same method of definitive screening was done

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for the present fuels of neem oils, and it was observed that an incremental of 5wt% gives maximum yield. Kapoor et al. (2020) investigated the performance and emissions of compression ignition engines with neem methyl ester mixed with cerium oxide nanoparticles. The results revealed that nanoparticle mixed blends of neem methyl ester and diethyl ester enhance performance and reduce harmful emissions.

Many conversion methods such as isomerisation and hydrotreatment, transesterification and Fischer-Tropsch have been reported in literature (Sajjad et al., 2014; Fasakin, 2016). The most effective of these processes is the transesterification reaction. Due to its simplicity, the transesterification reaction causes oil to react with alcohol in the presence of a base catalyst or an acid to yield glycerol and ester. It is used commercially in developed countries (Germany, France, Canada, Italy, New Zealand, etc.) and developing countries like Brazil and India (Konada et al., 2020). In some major oil-exporting countries, biodiesel generates high foreign exchange savings. In addition, it has recently become more attractive due to its environmental benefits, renewability, biodegradability, non-toxicity and cost-effectiveness.

Interestingly, it is also promoted as a technical option for responding to climate change. However, a wide range of feedstock (edible, in-edible oil crops and waste oils, as well as microalgae), diverse reaction and separation conditions, and different types of catalysts make biodiesel manufacturing a complex system that requires not only empirical work but also the modelling research efforts. Some recent review papers in literature provide a very good overview of the research advancement in biodiesel production, including feedstock resources and characteristics, oil extraction and transesterification methods, reactor design and process intensification (Muhammad et al., 2021; Bhatia et al. (2021) and Ananthi et al., 2021).

Many researchers used cooking oils such as melon and palm oil to make biodiesel. Still, the fluctuating prices of edible oils and increasing demands for nutritional needs

have made Neem Seed oil the raw material of choice. To overcome the earlier mentioned issues, engineers need to produce biodiesel using second-generation vegetable oil and a catalyst that reduces energy requirement in transesterification and is cost-effective. However, biodiesel and bioethanol alone cannot be used as an effective alternative fuel for diesel engines. Even diesel-biodiesel blends or diesel-bioethanol blends will not fulfil the requirements of these engines. Therefore, this study seeks to generate a ternary blend fuel which comprises a mixture of diesel, biodiesel and bioethanol that can be used in diesel engines with agreeable proportion.

Nevertheless, this paper seeks to perform an analysis of oxidative degradation and the characterisation of blends of diesel, bioethanol and neem seed-oil methyl ester as a fuel for diesel engines. The objectives are summarised as follows:

1. To characterise the biodiesel and bioethanol produced to ascertain its physiochemical properties by testing for properties such as kinematic viscosity, flash point and density.
2. To monitor the oxidative degradation process of ternary blends associated with structural and compositional changes using spectrometric methods.

THEORIES AND DEFINITIONS

Biodiesel feedstocks

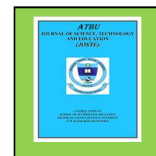
The feedstock used for biodiesel production varies with location according to climate and availability. The most abundant commodity oils or fats in a particular region are the most common feedstock. Each biodiesel feedstock has a unique chemical composition; biodiesel produced from different feedstock will, in turn, have different fuel properties (Bryan, 2010). There are several biodiesel feedstock categories: edible oilseeds, non-edible oilseeds, algae, animal fats (e.g., chicken fat, poultry fat and beef tallow), and various other waste materials such as used cooking oils and greases. Literature has shown that oilseeds are one of the significant renewable feedstocks that can provide an abundant,

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cost-effective and clean source of energy for biodiesel production. Common edible oilseeds include palm oil, groundnut oil, cottonseed, sunflower and soybean (Alamu *et al.*, 2007).

However, a common oil seed that can be used as biodiesel feedstock is neem seed (*Azadirachta indica*). Neem trees are abundant, typically in tropical and semi-tropical regions. The neem tree is popular in India, Indonesia, Malaysia, Myanmar, Pakistan, Senegal, Sri Lanka, and Thailand. It has since been transplanted to many parts of the world, including several countries in Africa, South America, Latin America, the Caribbean, the Middle East, and others (Martín *et al.*, 2010). Neem seeds obtained from this tree are collected, de-pulped, sundried and crushed for oil extraction. It comprises mainly triglycerides and large amounts of triterpenoid compounds, which are responsible for the bitter taste. It also contains azadirachtin, meliantriol, salannin, nimbinin, nimbin and nimbidin. The neem seed has a high oil content of 39-60% and also a large percentage of monounsaturated fatty acids (Martín *et al.*, 2010). In addition, it possesses a low proportion of polyunsaturated acids and a controlled amount of saturated fatty acids (Wang *et al.*, 2011).

Another good feedstock is Bioethanol (C_2H_5OH), an oxygenated biofuel produced from agricultural feedstocks. Bioethanol is primarily categorised into; first-generation feedstock (food, e.g., sugarcane, corn, cassava etc.), second-generation feedstock from non-edible lignocellulosic materials (either residue of grasses or trees) grown specifically for biofuel purposes or food crop production (e.g., corn stalks, cassava peels or rice husks). First generation feedstocks are divided into sugar and starch sources. At the same time, sugar-based raw materials include sugarcane, sugar beet and sweet sorghum, fruits (like grape, dates, watermelon and apple) and the sugar refinery waste, namely cane molasses and beet molasses (Nwufu *et al.*, 2016). These materials contain sufficient organic nutrients and readily fermentable sugars,

making them an ideal raw material for bioethanol production (Zabed *et al.*, 2016).

METHODS OF BIODIESEL PRODUCTION

Micro-emulsion of oils

Micro-emulsion of oils occurs when a colloidal equilibrium dispersion of optically isotropic fluid with dimensions generally in the 1-150 nm range forms spontaneously from two normally immiscible liquids and one or more ionic or non-ionic amphiphiles (Slomkowski S. *et al.*, 2011). Biodiesel micro-emulsion consists of diesel fuel, vegetable oil, alcohol, and surfactant and cetane improver in suitable proportions. Alcohols such as methanol and ethanol are used as additives for reducing viscosity, higher alcohols are used as surfactants, and alkyl nitrates are used as cetane improvers (Chiaromonti *et al.*, 2003). Micro-emulsions containing butanol, hexanol and octanol can ascertain the maximum viscosity requirement for diesel fuel. For example, 2-octanol was found to be an effective amphiphile in the micellar solubilisation of methanol in triolein and soybean oil (Schwab *et al.*, 1987).

Pyrolysis (Thermal cracking)

Pyrolysis involves heating chemical bonds in the absence of air and cleavage to produce tiny molecules. Also, it encompasses the conversion of one substance into another with the aid of a catalyst. The pyrolysed material can be vegetable oils, animal fats, natural fatty acids and methyl esters of fatty acids. It has been observed that the pyrolysis of triglycerides to obtain biodiesel is suitable for diesel engines. The liquid fractions of the temperature-based conversion of vegetable oils will likely approach diesel fuels. This type of decomposition of triglycerides produces alkanes, alkenes, alkadienes, aromatics and carboxylic acids (Atabani *et al.*, 2012; Balat M. *et al.*, 2010). Low viscosity, flash point and pour point were observed in the pyrolyzate than in petroleum diesel fuel (Atabani *et al.*, 2012). Singh and Singh (1991) reported that the pyrolysis process is effective, simple, zero waste, and pollution-free. The cetane number (CN) of the pyrolyzate vegetative oil

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or fat was lower. Pyrolyzed vegetable oils have an acceptable quantity of sulphur, water and sediments and give acceptable copper corrosion values but unacceptable ash, carbon residual, and pour point.

Transesterification of oils and fats

According to Sharma *et al.* (2008), transesterification is the process of using alcohol, like methanol or ethanol, in the presence of sodium hydroxide (NaOH) or potassium hydroxide (KOH) catalyst to chemically break the molecule of the raw renewable oil into methyl or ethyl esters of the oil with glycerol as a by-product. It is the most popular method used for biodiesel production. It consists of three consecutive reversible reactions where triglycerides are converted to diglycerides. Then diglycerides are converted to monoglycerides, followed by the conversion of monoglycerides to glycerol. In each step, an ester is produced, and thus three ester molecules are produced from one molecule of triglycerides.

Related works in literature

Many researchers have reported that biodiesel and traditional diesel have indistinguishable physical and chemical properties (Alptekin & Canakei, 2008; Murillo *et al.*, 2007; Nabi *et al.*, 2006; Sharma *et al.*, 2005). Datta *et al.*, 2016 presented a comprehensive review of biodiesel as an alternative fuel for compression ignition engines. They concluded that the environmental aspects improved by using biodiesel, but the engine performance deteriorated greatly. Vegetable oil and animal fat are used to make biodiesel, which is non-toxic. High cetane and no sulfur are two benefits of biodiesel. However, the main drawback was that ordinary diesel fuel could not be completely replaced. Another concern is the fact that biodiesels are characterised by low calorific values but high Kinematic viscosity and density values (Sharp *et al.*, 2005). Moreover, reports from many research studies that blended biodiesel with diesel at various proportions, have shown that the calorific values of biodiesel were improved while engine performance was increased (Suryawanshi & Deshpande,

2005; Sirman *et al.*, 2000; Krahl *et al.*, 1996; Rao *et al.*, 1991; Rakopoulos *et al.*, 2005; Agarwal & Das, 2001; Rosea *et al.*, 2005; Kumar *et al.*, 2001; Rao *et al.*, 2005).

Gad *et al.* (2020) studied the effect of cotton ethyl ester blend enriched with HHO (2 hydrogen and one atom oxygen molecules) gas and kerosene additives on a diesel engine's performance and emissions characteristics. The results show that adding a kerosene additive enriched the cold flow properties. On the other hand, HHO inclusion enhanced combustion characteristics. Adu-Mensah *et al.* (2019) reviewed the developments associated with hydrogenated biodiesel to improve the oxidation stability without compromising the cold flow properties. They reported that using a mixture of catalysts could reduce the trans-FAME (Fatty Acid Methyl Esters) and partial hydrogenation. Chandran (2020) reviewed the compatibility of biodiesel with fuel delivery materials (FDM) and the FDM degradation due to exposure to oxidised biodiesel containing oxidised products such as aldehydes, ketones and short-chain acids was not true for CRDE (Common Rail Diesel Engine). Still, biodiesel's dissolved oxygen and conductivity value have an adverse effect. Jiaqiang *et al.* (2017) comprehensively investigated various technologies applied to biodiesel-fueled engines. They found the Low-Temperature Combustion (LTC) mode is more efficient in reducing the NO_x and PM emissions when compared to other technologies like biodiesel additives, exhaust gas recirculation (EGR), water injection (WI), emulsion technology (ET), injection strategy modification, and combustion chamber geometry modification. Wang *et al.* (2016) reviewed the particulate matter emission characteristics of diesel engines with biodiesel or biodiesel blends. They highlighted that the properties of biodiesel fuel, such as the fatty acid chain length, the degree of unsaturation and oxygen content, greatly affect the particulate matter emission.

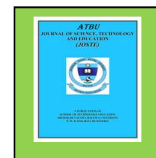
Akhabue *et al.* (2020) embedded sulfonated calcined corncobs and calcined poultry droppings to prepare a novel bio-based bifunctional catalyst (BBFC). The neem seed

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oil (NSO) contains free fatty acid content of 4.426% treated with the bio-based catalyst. Ramalingam et al. (2020) investigated the effects of *moringa oleifera* biodiesel blend using sophisticated injection strategies on the diesel engine properties. They observed that timings (15°CA to 25°CA bTDC) and the fuel injection pressure (300 bar to 600 bar) greatly influenced engine emissions. Their experiment showed that adding *moringa oleifera* biodiesel blend significantly reduced CO and CO₂ emissions. Suresh et al. (2018) reviewed previous researchers' results using a variable compression ratio (VCR) engine with direct injection of biodiesel blend fuel. They suggested that a VCR engine with optimum compression pressure enhances the engine performance and reduces the harmful emission gases. Singh et al. (2019) have reviewed the chemical compositions, properties, and standards for various biodiesels. They reported that modifications to the FAME's compositional component had an impact on the chemical and physical properties of biodiesel. Both beneficial and undesired changes in FAME characteristics would result from variations in chain branching, unsaturation, and chain length. Poor performance at low temperatures may result from the compositional properties of FAME that improve fuel stability. It is challenging to determine a precise fatty acid composition since the content of fatty acids reversely affects the properties of biodiesel. Its cold flow, cetane number, lubricity, viscosity and oxidative stability are the qualities that show that FAME is a suitable feedstock for biodiesel. Because they fluctuate depending on the feedstock, oxidation stability and cold flow are two of these qualities that are crucial. However, less polyunsaturated fatty acid value, which reduces oxidative instability, is an indicator of high-quality FAME.

Mirhashemi et al. (2020) reviewed the potential of various biodiesel blends such as gasoline, hydrogen, natural gas, biogas, and various alcohol types to reduce NO_x emissions. Additionally, they stated that the correct fuel blending proportions and injection parameters must be specified to lower NO_x emissions. Erdiwansyah et al.

(2019) presented a paper that contrasted the effect of alcohol, biodiesel and gasoline on engine performance. They also evaluated the combustion and emission properties of each fuel source. Furthermore, they identified the merits and demerits of using biodiesel and alcohol in engines. Moreover, Yesilyurt et al. (2020) studied the performance, combustion, and emissions properties of diesel engines operating on a mixture of diesel fuels, biodiesel, and 1-heptanol (C7 alcohol) as the next generation higher alcohol. They found that adding 1-heptanol as an oxygenated additive to diesel fuel and biodiesel/diesel fuel mixtures resulted in a reduction in CO and unburned HC emissions compared to diesel fuel, CO₂, O₂ and NO_x.

Shahir et al. (2015) examined the possibility and prospects of using a diesel-biodiesel-ethanol / bioethanol mixture in an existing diesel engine. They reported that keeping the ethanol content below 5% gave better results. However, they added that reducing NO_x emissions remains challenging, and additional catalytic converters must be installed to control it. Zaharin et al. (2017) studied the effect of biodiesel blending with alcohol on diesel engine performance and exhaust gas. They noticed that adding alcohol mixtures reduces CO, HC, and PM emissions and increases NO_x emissions. Hasan et al. (2017) studied the environmental and economic impacts of biodiesel production, the impact of biodiesel blends on internal combustion engine life, performance and emission characteristics. They concluded that a small portion of the biodiesel mixture could be delivered to an existing engine without modification. Consequently, few engine modifications are mandatory for a higher percentage of biodiesel and long-term use. Soudagar et al. (2018) outlined the disadvantages of using biodiesel. These included increased nitrogen oxide emissions, an inability to operate in cold weather, and a need to repair engine equipment such as fuel filters, fuel tanks, and fuel lines at regular intervals resulting from clogging. They suggested that adding nanoparticles as fuel additives could improve the properties of biodiesel. The addition of

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metallic nanoparticles resulted in a reduced ignition delay period and enhanced the calorific value and oxidation rates leading to a complete and cleaner combustion. Guo et al. (2020) investigated the effects of diesel, xylene, butanol, coconut oil and toluene on the diesel engine's thermal oxidation and chemical structure. They concluded that thermal oxidation and the chemical structure of fuels vary depending on the fuel identity. Biodiesel's soot is more reactive for more mass loss at a lower temperature range.

MATERIALS AND METHODS

The major raw materials used in this study are neem seeds and cassava peels. The neem seeds were obtained from the Maraba market, while the cassava peels were obtained from a cassava processing site at Masaka, Nasarawa State, Nigeria.

Saccharomyces cerevisiae (Baker's yeast) and *Aspergillus niger* were cultured in Nile University microbiological lab. However, other materials used include; sulphuric (H_2SO_4) acid, methanol (99.5% purity), petroleum ether, distilled water, chloramphenicol (250mg) capsules, potassium hydroxide (KOH) and hydrochloric acid (HCl).

Moreover, the equipment employed during the experimentation performed in this study are; a round-bottom flask, Petri-dishes, incubator, heating mantle, microscope, Soxhlet extractor, stopwatch, magnetic stirrer, thermometer, 500ml Erlenmeyer flask, 500ml Separatory funnel, Viscometer, Flash Point Tester, UV-Spectrometer and FTIR Spectrometer. The summary of the methodology used in this study is illustrated in Figure 1.

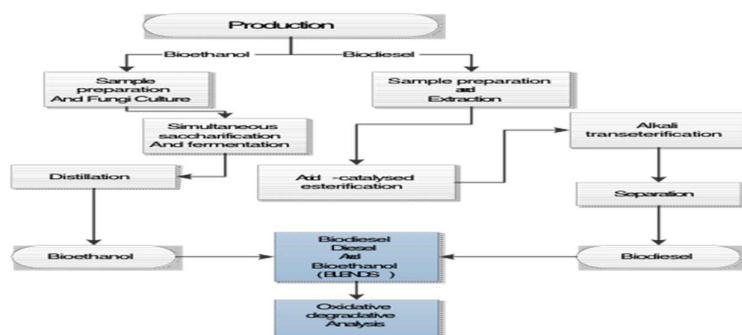


Figure 1: Methodology Flowchart

PRODUCTION OF BIODIESEL AND BIOETHANOL

Neem Seed Preparation and Oil Extraction

Prior to use, the neem seeds were washed thoroughly to remove dirt and other impurities material to avoid contamination. The seed was sun-dried for a week in order to remove water content and increase oil yield. The dried seeds were crushed in a grinder in order to reduce the particle size as well as increase the surface area, which in turn increases the oil yield. A solvent extraction method using petroleum ether was adopted. The dried, powdered neem seeds were weighed and placed in the Soxhlet apparatus for 3 hours with a ratio of neem seed powder weight to solvent volume of 1:5 (w/v) to allow proper diffusion of oil into the

solvent. The solvent-oil mixture was then separated from the solution using the rotary evaporator to recover the solvent. The extracted oil was then left to enable evaporation of any remaining amount of petroleum ether to obtain solvent-free oil and later stored in an air-tight waterproof container which was placed in a cool, dry place for further analysis.

Production of Biodiesel from neem oil

Biodiesel from the neem oil extracted was produced through the transesterification process in three steps. In order to reduce the high amount of fatty acids in the oil, the neem oil undergoes pretreatment so as to avoid the formation of soap.

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Acid-catalysed Esterification

The esterification was carried out using 0.40 v/v, i.e. 400ml of methanol and 1% v/v, i.e. 10ml of concentrated H₂SO₄ mixed together inside a 2000ml round-bottom flask. The round-bottom flask was inserted into a water bath at 50°C. The 1000ml of warmed (preheated) neem oil and 400ml of methanol, H₂SO₄ mixture was taken in a 2000ml container and placed on a hot plate, continuously stirring at 500rpm using a magnetic stirrer for 1 hour at 60°C for the reaction to take place. After the esterification, the mixture was allowed to settle for 12 hours. The top layer consisting of the esterified oil and the bottom layer known as residues was obtained. The bottom layer was removed, and the esterified oil was used for the transesterification reaction (See Figure 3.2).

Alkali Transesterification

0.30 v/v (i.e. 240ml) of methanol and 0.5% w/v (i.e. 4g) of KOH as catalyst

were dissolved in a conical flask to form a methoxide solution. This was then added to the esterified oil. The mixture was heated on a hot plate and continuously stirred at 500rpm for 1 hour at 60°C for the transesterification process to take place. The biodiesel was allowed to settle for at least 6 hours in a separating funnel to allow clear separation of the biodiesel from glycerol. The biodiesel layer was separated by decantation as the glycerol was drained off from the separating funnel.

To wash and dry the biodiesel, warm distilled water at 50°C was added to the collected biodiesel, and the mixture was slightly shaken. The water was then drained through the bottom of the separating funnel. This was carried out three times until clear biodiesel was obtained. The distilled water helps remove traces of glycerol and free impurities.



Figure 2: Separation of Biodiesel (Upper Layer) and Glycerol (Lower Layer)

PRODUCTION OF BIOETHANOL

Inoculation of Fungi (*Aspergillus niger* and *Saccharomyces cerevisiae*)

The pure cultures of *Aspergillus niger* and *Saccharomyces cerevisiae* were inoculated from soil samples and baker's yeast, respectively. 150ml of distilled water and 500mg of chloramphenicol were added to 15g of nutrient agar and allowed to sterilise in an autoclave for 15 minutes. Pure culture media were sub-cultured in different Petri-dishes. The media was kept for fourteen (14)

days in an incubator and temperature of 30°C.

Preparation of Cassava peels and Broth culture of *A. niger*

The cassava peels were hand-peeled from cassava tubers using a table knife. The peels were washed to remove sand and other impurities. The dried peels were then air-dried under the sun for seven (7) days, after which it was milled into a powder (flour) using a locally made grinder and sieved with 1.5 μ aluminium sieve. The

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flour was packed into polythene bags and sealed until further use. Broth culture of *Aspergillus niger* was prepared in 100ml of nutrient broth medium using standard methods as described by Baker *et al.* (2001).

Simultaneous Saccharification and Fermentation

Fifty grams of the sieved cassava peel flour was weighed into three conical flasks and dissolved in 500ml of distilled water. The flasks were plugged with sterile cotton wool, shaken thoroughly and autoclaved for 15 min at 120°C (Adesanya *et al.*, 2008). Each of the six flasks were inoculated with 5ml *A. niger* + 7g of *S. cerevisiae*. The mixture in each conical flask was sealed with an aluminium foil and kept for twenty-eight (28) days under anaerobic conditions and a temperature of 30°C. Thereafter, the samples were filtered with Whatman No. 3 filter paper, and 300ml of the filtrate was distilled at 78°C (standard temperature for ethanol distillation). This was done for each fermented sample.

CHARACTERISATION OF BIODIESEL AND BIOETHANOL

Determination of Flashpoint

The sample was placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover was then fitted onto the position on the cup, and a Bunsen burner was used to supply heat to the apparatus at a rate of about 5°C per minute. During heating, the biodiesel/bioethanol was constantly stirred. As the oil approached its flashing, the injector burner was lighted and injected into the

container every 15-second intervals until a distinct flash was observed within the container. The temperature at which the flash occurred was then recorded.

Determination of Viscosity

A viscometer was inserted into a water bath with a set temperature of 40°C and left for 30 minutes. The sample was then added to the viscometer and allowed to remain in the bath so that it reaches the test thermometer. The sample was allowed to flow freely, and the time required for the meniscus to pass from the first to the second timing mark was taken using a stopwatch.

Determination of Density

An empty calibrated measuring cylinder, 50ml was weighed, and 20cm³ of the biodiesel/bioethanol sample was introduced. The cylinder containing the biodiesel/bioethanol was then weighed. The mass of empty cylinder was subtracted from the mass of the cylinder containing the biodiesel/bioethanol sample to find the mass of the biodiesel/bioethanol sample.

The density of the sample was calculated by dividing the mass by the volume.

BLENDING

Blending was done for each biodiesel with bioethanol and fossil diesel of 50ml in a ratio of 5:5:95, 20:10:70 and 40:30:30. These blends were denoted as (BD5BE5D90), (BD20BE10D70), and (BD40BE30D30) respectively. The blends were stored in reagent bottles for a period of 21 days for further oxidative degradation analysis (See Figure 3).



Figure 3: Biodiesel-Bioethanol-Diesel Blends

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OXIDATIVE DEGRADATION ANALYSIS
Ultraviolet-visible Spectroscopy (UV-Vis)
 Three blends of bioethanol-biodiesel-diesel were measured in different proportions; (BD5BE5D90), (BD20BE10D70) and (BD40BE30D30). The

absorbance readings of each ternary blend were obtained using a Perkin-Elmer Lambda 25 UV/Vis spectrometer in the wavelength range of 190-800nm with a quartz cuvette at room temperature and diesel was used in blank cell.



Figure 4: Ultraviolet-Visible Spectrometer for Absorbance of Blends

Fourier Transform Infrared Spectroscopy (FTIR)
 The infrared spectra for the three blends were obtained using a Perkin Elmer Spectrum 400 FTIR spectrometer in the wavenumber range of 4000–500 cm^{-1} with 2 cm^{-1} resolution and three scans at room temperature.

obtained for the biodiesel, bioethanol and ternary blends produced. Results for oxidative degradation from UVVis and FT-IR are also discussed in this section.

RESULTS AND DISCUSSION

The values for relative density, flashpoint and kinematic viscosity were

Characterisation of Biodiesel and Bioethanol Produced

The values for relative density, flashpoint, and kinematic viscosity obtained for the biodiesel and bioethanol produced are in Table 4.1.

Table 4.1: Physiochemical properties of Biodiesel and Bioethanol Produced

Properties	Biodiesel	Bioethanol
Relative density (g/cm^3)	0.871	0.854
Viscosity (mm^2/S)	4.2	----
Flashpoint ($^{\circ}\text{C}$)	136.0	28

Density is a fuel-property that has direct effects on the engine performance characteristics. The density obtained for the biodiesel and bioethanol are $0.871\text{g}/\text{cm}^3$ and $0.854\text{g}/\text{cm}^3$ is within the specified value by the EN ranges from 0.860 to $0.900\text{g}/\text{cm}^3$ for biodiesel and bioethanol. Kinematic viscosity

for biodiesel was determined to be 4.2mm^2 that falls within the recommended EN limit of biodiesel. This will enhance the flow property of the biodiesel in the diesel engines. Transesterification helps to lower the kinematic viscosity to prevent the formation of large droplets on injection that can cause

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poor combustion, increased exhaust and emissions. The flash point for the biodiesel and bioethanol produced was determined to be 136°C and 28°C respectively, which is in agreement with EN specification standards of biodiesel and bioethanol. The flash point determined for the produced biodiesel can reduce the extent of problems such as fire

hazards, which can be caused by the lower flash point of fuel.

Characterisation of Biodiesel-Bioethanol-Diesel (BD-BE-D) Blends

The values for relative density, flashpoint and kinematic viscosity obtained for the biodieselbioethanol-diesel (BD-BE-D) produced are in Table 4.2.

Table 4.2: Physiochemical properties of Biodiesel-Bioethanol-Diesel Blends

Blends	Density (g/cm ³)	Viscosity (mm ² /S)	Flash Point (°C)
BD5BE5D90	0.863	3.72	17
BD20BE10D70	0.869	3.83	28
BD40BE30D30	0.878	4.23	51

The density of the blends increases as the percentage of biodiesel increases in the blends that is attributed to the fact that biodiesel has a high density, which increases the final density of the blends. The percentage of bioethanol in the blends does not affect the final density due to its low density. The flash point of these blends (biodiesel–diesel–bioethanol) is mainly dominated by bioethanol. With flash point values ranging from 51°C-17°C, proper precautions must be taken for storage, handling and transporting the fuel. Kinematic viscosity increases with an increase in the percentage of biodiesel as it seen in Table 4.2. All values fall within the range of EN specifications for ternary blend fuels and ASTM standards for diesel fuel.

vs. transmittance percentage (%T) in Fig. 3.1, Fig. 3.2 and Fig. 3.3 by plotting the regions of infrared spectra which have been transmitted by specific molecular bonds presenting in the sample blends. A higher percentage of infrared transmittance in the graph symbolises the lower presence of respective molecular compounds in the samples. Wave number ranges 3000–3700 cm⁻¹, 2700–3000 cm⁻¹ and 1755-1650cm⁻¹ represents O-H, C-H and C=O bonds respectively. As can be seen from Fig. 3.1, the intensity of the ester carbonyl C=O stretching peak noticeably at 1751 cm⁻¹ as a result of biodiesel, and also H-C-H band at 2853-2954 cm⁻¹. The peak at 1459 cm⁻¹ shows the presence of benzene due to the percentage of diesel in the blend. The O-H bond is significantly not noticeable due to the presence of bioethanol in this spectrum. This shows that degradation has occurred during the period of storage.

OXIDATIVE DEGRADATION ANALYSIS FTIR Data Interpretation

FTIR spectrum data for all samples has been displayed as wave number (cm⁻¹)

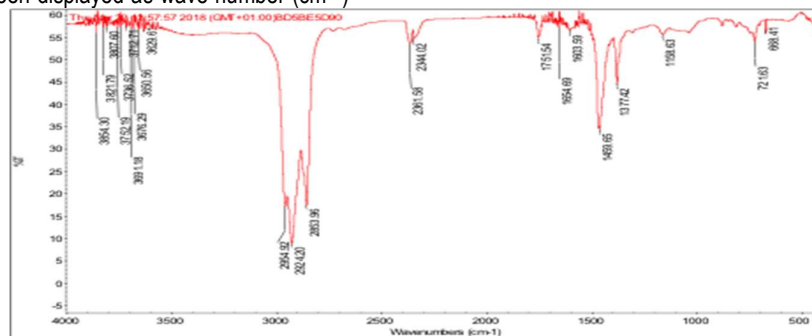


Figure 3.1: FTIR Spectrum for BD5BE5D90 Blend

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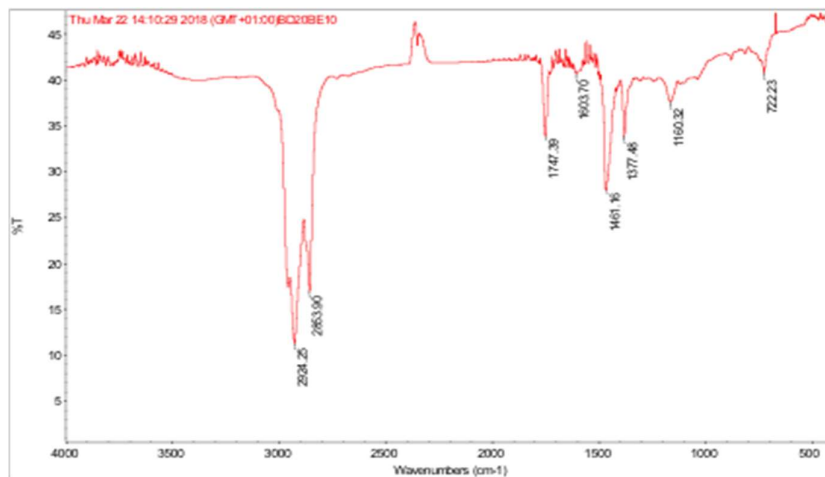


Figure 3.2: FTIR spectrum for BD20BE10D70 Blend

Moreover, the spectrum in Fig. 3.2 shows the presence of C=O at 1747.39 cm^{-1} due to the presence of biodiesel. There is no noticeably O-H band at 3320 cm^{-1} due to the presence of bioethanol in the blend. In addition, a peak can be seen within the range of $2800\text{--}2900\text{ cm}^{-1}$ which is due to the

presence of H-C-H bonds in diesel. This shows that degradation has occurred during the period of storage. Formation of primary oxidation products, hydroperoxides, which are unstable and can further break down to form secondary oxidation products, such as ketones, epoxides and oligomers.

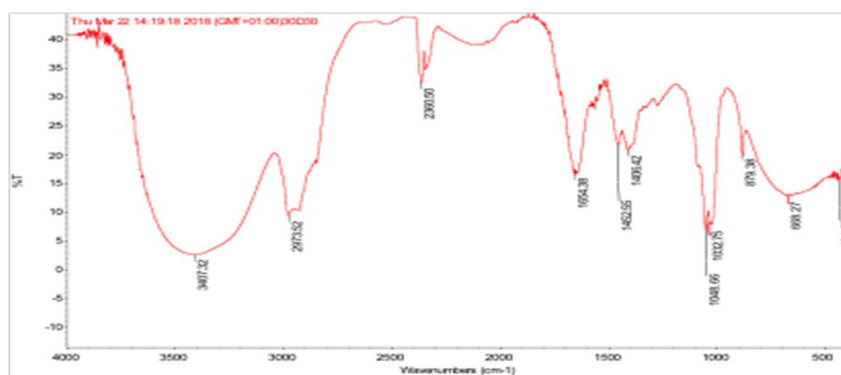


Figure 4.3: FTIR Spectrum for BD40BE30D30 Blend

Fig. 3.3 spectrum shows the presence of a broad peak at 3407 cm^{-1} , indicating the presence of an alcohol group (O-H) with a noticeably stretching ester (C=O) peak at 1654 cm^{-1} , indicating the presence of biodiesel. The peaks at 2973 cm^{-1} and 1452 cm^{-1} indicates the presence of CH sp³ in diesel fuel. This blend has not undergone any form of degradation during the period of storage.

UV-Vis Data Interpretation

According to the UV-Vis spectroscopy result, this technique shows the indication of oxidative degradation of biodiesel-bioethanol-diesel blends. Figure 4.4 shows that UV-Vis spectrum for the blend samples; (BD5BE5D90) and (BD20BE10D70) presented absorption maxima at 230 nm with 8.0 and 4.69 respectively. This behaviour occurs when the oxidation of polyunsaturated fatty acids is accompanied

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by displacement of isolated double bonds for conjugated double bonds. Sample BD40BE30D30 has significantly low

absorption value 3.8 at 230 nm which indicates low degradation.

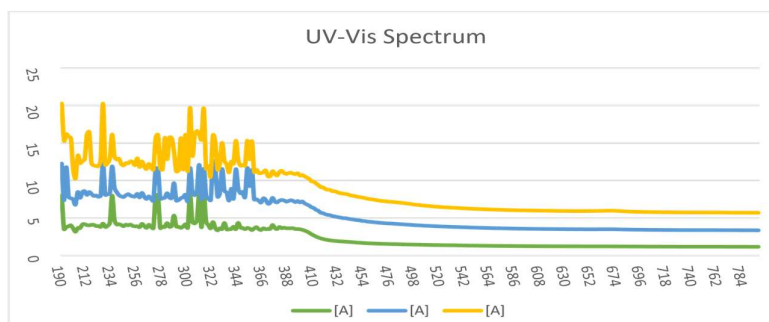


Figure 3.4: UV-Vis Absorption Spectrum for BD5BE5D90, BD20BE10D70 and BD40BE30D30

CONCLUSION

Degradation of biofuels alters fuel properties which include increase in viscosity, density, heating value, iodine value and flash point. Therefore, it presents a legitimate concern in maintaining the fuel quality during storage and use. Ternary blends (bioethanol-biodiesel diesel) are generally resistive to oxidative degradation but usually depends on the exposure to favorable conditions such as air and certain metals can degrade the stability of the blend. BD5BE5D90 and BD20BE10D70 have low flash point which lead to explosion in high temperature; also the spectrometric analysis shows that these blends can degrade overtime during storage. On the other hand, BD40BE30D30 showed a better result with physicochemical properties which are within the standards of the EN specification of fuel. The FTIR and UVVis analysis also shows that the blend has not undergone oxidative degradation. Therefore, BD40BE30D30 is a promising eco-friendly biofuel blend with sustainable development.

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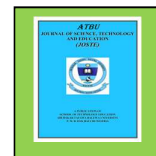
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