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Optimization of Biodiesel Yield from Neem Seed Oil Using Calcined Eggshell Catalyst: Characterization and Performance Analysis

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Abstract

This study examines the optimization of biodiesel yield production from Neem seed oil (Azadirachta indica *A. Juss), a non-edible and renewable feedstock. The aim is to develop a sustainable, environmentally friendly fuel alternative using calcined eggshell as a low-cost heterogeneous catalyst in a base-catalyzed transesterification process. Catalyst concentrations of 0.5%, 1%, and 1.5% (wt.% of oil) were tested at reaction times of 70, 110, and 150 minutes, yielding nine biodiesel samples. The optimum biodiesel was analyzed for physico-chemical properties, while GC-MS and FTIR were used to determine its chemical composition and functional groups. The optimum sample was blended with diesel to form B10, B20, and B30. These fuels were tested on a single-cylinder CI engine at a constant load of 4.5 kN and 1500 rpm to evaluate performance and emissions. A maximum yield of 100% was obtained at 1% catalyst concentration and 70-minute reaction time. B30 and B100 demonstrated the highest brake thermal efficiency, while B10 recorded the lowest exhaust gas temperature. Brake power increased with higher biodiesel content, with B30 and B100 giving the best results. Emission analysis showed lowest UHC, NOx, and CO_2 emissions at B20 and B30, while CO emissions were zero for all blends. Neem-based biodiesel produced with calcined eggshell catalyst is a viable and cleaner alternative to petro-diesel, offering strong performance and reduced emissions. Further research should examine catalyst reusability, scale-up feasibility, multi-load engine testing, long-term engine durability, and ethanol-based transesterification.*

Keywords: *Biodiesel, neem seed oil, transesterification, eggshell, calcination, catalyst, characterization.*

1. **Introduction**

The growing environmental pollution associated with the continuous use of conventional fossil fuels has intensified the search for sustainable and cleaner fuel alternatives for compression ignition (CI) engines. Fossil fuel combustion, particularly in the transportation sector, accounts for a significant share of global greenhouse gas emissions—contributing about 25% of atmospheric GHGs and up to 59.5% of CO_2 emissions in Nigeria. This challenge has motivated researchers to explore renewable fuels capable of reducing emissions while maintaining engine performance (Ben-Iwo et al., 2016; May & Marsden, 2010).

Biodiesel has received global attention as a promising substitute due to its renewable nature, biodegradability, and improved combustion characteristics. However, the direct use of vegetable oils in CI engines is limited by high viscosity, poor volatility, and the risk of incomplete combustion, which can damage engine components. To overcome these drawbacks, chemical modification processes such as transesterification are employed, with transesterification being the most effective method for reducing viscosity and meeting standard fuel properties (Miyuranga et al., 2023).

Although edible vegetable oils have traditionally been used for biodiesel production, concerns over high cost, food security, and land degradation make them unsuitable for large-scale applications. This has shifted attention to non-edible oils such as Neem, Jatropha, castor, and algae oils. Neem seed oil, in particular, is abundant in tropical regions like Nigeria and remains underutilized despite its significant production potential (Wan Ghazali et al., 2015).

Catalyst selection is critical in biodiesel production, and calcium oxide (CaO) is widely preferred due to its strong basicity and low cost. Recent studies have shown that waste eggshells, rich in calcium carbonate, can serve as an inexpensive and effective source of CaO when calcined at high temperatures. This approach not only lowers production costs but also supports environmental sustainability through waste-to-resource conversion (Shan et al., 2016).

This study therefore aims to produce economically viable biodiesel from non-edible Neem seed oil using calcined waste eggshells as a heterogeneous CaO catalyst, while evaluating the fuel's physicochemical properties and chemical composition for potential commercial application.

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2. **Literature Review**

The increase in environmental pollution partially resulted from the burning of conventional fuels over time, challenged researchers to investigate and look for reliable fuel substitute for CI engine. Utilization of fossil fuels in IC engine, particularly in an automotive transportation section is part of the scary trouble to our present time societal development because it contributes GHG of about 25% to the atmosphere (May & Marsden, 2010). As CI engine having a big portion in the leading fill of the automotive transportation section, various academicians became enticed to study in the aspect of supplementary or alternative CI engine's fuel (Datta & Kumar, 2016). Petroleum consumption for road transportation is currently the largest source of CO_2 emissions. It accounts for 23% of CO_2 emissions worldwide and 59.5% of CO_2 emissions in Nigeria (Ben-Iwo et al., 2016).

Scientific community and researchers worldwide have focalise on the exploitation of biodiesel and the process optimization to accommodate the acceptable specifications required for the commercial utilization of the fuel with no compromised concerning the longevity of engine parts. The curiosity for the utilization of renewable energy particularly fuel commenced with the direct replacement of diesel with vegetable oils. Nevertheless, high viscosity limited the direct use of vegetable oils in CI engines due to its negative effect such as insufficient atomization, partial combustion and engine failure caused by accumulation of carbon on the injector and choke support (Ramadhas et al., 2005). Additional restrictions for the usage of unmodified vegetable oil were polyunsaturated behavior and low volatile nature of the oil. To get over the aforementioned restrictions, modification method such as micro-emulsification, pyrolysis, trans-esterification, etc. were specially initiated. The systematic chemical reaction between triglycerides and alcohol with the aid of catalyst is termed as trans-esterification. This process lie on three successive series of reversible reactions to which triglycerides are transformed to diglycerides, diglycerides to monoglycerides and monoglycerides is finally converted to glycerol. An ester is produced at each transformation stage, thereby producing three molecules of ester from single molecule of triglyceride (Sharma and Singh, 2007).

$$Triglyceride(TG) + ROH \leftrightarrow Diglyceride(DG) + RCOOR1 \quad (1)$$

$$Diglyceride(DG) + ROH \leftrightarrow Monoglyceride(MG) + RCOOR2 \quad (2)$$

$$Monoglyceride(MG) + ROH \leftrightarrow Glycerol + RCOOR3 \quad (3)$$

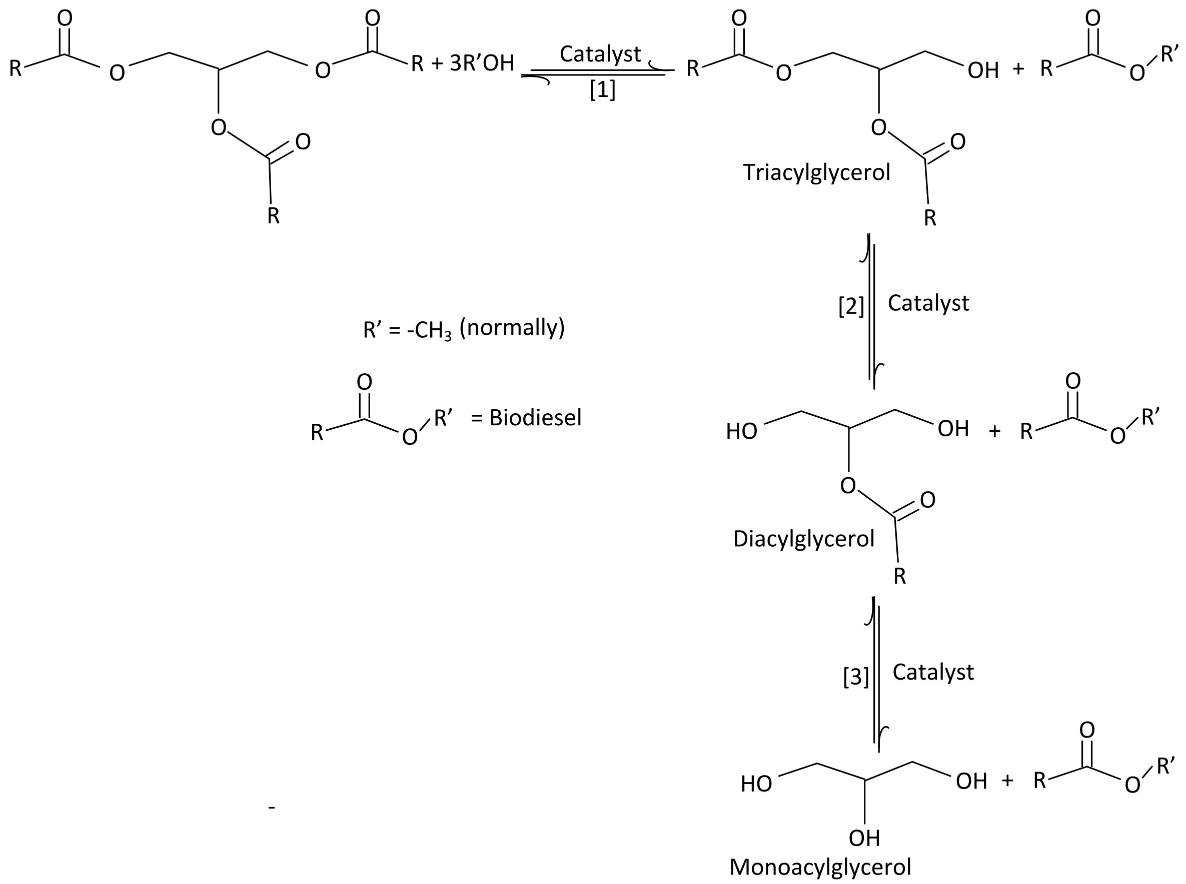

Figure 1: Transesterification reaction of triacylglycerol

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From the three-vegetable modification process mentioned earlier, trans-esterification happens to be the most viable and reliable method adopted up to present for viscosity reduction purposes (Sharma et-al, 2008).

Any type of organic compounds that contain at least one hydroxyl functional group bound to a saturated carbons atom is referred as alcohol. Some of them are butyl-alcohol, propyl-alcohol, ethyl-alcohol, methyl-alcohol, amyl-alcohol etc. The commonly used alcohols are methyl-alcohol and ethyl-alcohol otherwise called methanol and ethanol respectively. Though, the polar and short chain characteristics associated with the low-cost advantages of the methanol makes it more suitable for use. Triglycerides do also react with methanol rapidly and comfortably dissolve the alkali catalyst. Though, the low boiling point of the methanol makes it more vulnerable to explosion risk, and hence it should be use with caution while producing the biodiesel.

Edible oils from vegetable such as sunflower oil, peanut oil, palm oil, etc. are the conventional raw materials for the production of biodiesel. However, the cost of these raw materials run to more than 80% of total biodiesel production cost which clearly demonstrate that the cost is on the high side. In addition, price increase of edible vegetable oils, food insecurity and soil degradation due to large scale farming are very likely. These enabled many related agencies to conclude that the edible option is reliably impossible (Na et al., 2015). Non edible vegetable oil such as Neem oil, Jatropha curcas oil, castor oil, algae oil etc. could be considered as reliable feedstocks for the production of biodiesel in commercial scale (Huerga et al., 2014).

Among the available wild oils, Neem oil has the highest potential as well as production. It is used particularly in pharmaceutical and soap industries which covers only 20-25% of its total potential. However, about 75-80% of the neem oil potential to date are not even exploited and can be a better option for biodiesel production (Yadav et al., 2013). Neem oil can be regarded as vegetable oil pressed from the seed of neem (Azadirachta indica) and evergreen tree that is widely spread in the tropical Africa such as Nigeria (Banu et al., 2018).

Table 1. Physico-chemical properties of neem seed Oil (NSO)

Parameters	Value	Method
Density at 25°C(kg/m^3)	0.912	ASTM D 4052
Viscosity at 37.8°C(mm^2/s)	49.79	ASTM D 445
Flash point in °C	227	ASTM D 93
Pour point in °C	12	ASTM D 97
Freezing point in °C	10	ASTM D 97
Acid value in mg.g^{-1}	9.10	AFNOR T60-204
Iodine value in g.100g^{-1}	74.82	AFNOR T60-203
Saponification value in mg.g^{-1}	200.54	AFNOR T60-206
Calorific value in MJ.kg^{-1}	39.53	-
Peroxide value in meq O_2.kg^{-1}	1.49	AFNOR T60-220
Carbon residue (wt%)	1.45	ASTM D 189
Ash (wt%)	0.02	ASTM D 482
Sediments (wt%)	0.01	ASTM D 4052
Water content (v%)	<0.05	ASTM D 9590

(Djibril et al., 2015)

Right choice of catalyst is another important aspect in trans-esterification process, which influence the expenses incurred during production and under certain conditions translate into economic hinderance. Despite the advantages of heterogeneous solid base catalyst over homogeneous base catalyst, calcium oxide (CaO) is the most appropriate for trans-esterification reaction which is attributed to its high basicity and low production cost (Shan et al., 2016). Nearly all of the transesterification reactions have been given more

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attention on the usage of CaO catalyst from the conventional source for the production of biodiesel. Beside these conventional sources of CaO, many fact finders make good use of eggshells to produce CaO for oil transesterification. The eggshell which use to be trash is now look on as a good source of CaO. Households, hotels, restaurants and indomie joints are the major source of eggshells, they produce it in large scale which used to be an issue regarding to solid trash disposal. The eggshell well stocked in calcium carbonate ($CaCO_3$) can be transformed to a catalyst substantial in CaO through a process called calcination. In preparation prior to the calcination, the eggshells generated are clean and washed with clean water for the removal of the removal of the inner thin layer. Thereafter it will be dried under the sun for 2 days and finally placed inside a muffle furnace to be calcined at a temperature of 800^{o}C for 3hrs (Kavitha et al., 2019). Other findings revealed that, trash eggshells begin with thorough washed in clean water to get rid of sand and any other foreign matter therein. It is then oven-dried at a temperature of 110^{0}C for 40 minutes. After that, the internal thin linings of the eggshells will be removed diligently and the plain eggshells would be grinded into fine particles using electrical grinding machine. The powder particles size of 80μm would be separated from the initial fine particles through sieve separation method. Then, the calcination of the powder particle would be carried out in an electric furnace at a temperature of 850^{o}C for period of 4hrs to achieved smooth conversion of eggshell well stocked in $CaCO_3$ to catalyst substantial in CaO (Ayodeji et al., 2018).

3. MATERIALS AND METHOD

3.1 Materials

The materials to be used in this work are Neem seeds oil, egg shells, potassium hydroxide (KOH), sulphuric acid (H_2SO_4), propan-2-ol, phenolphthalein indicator, methanol, water and petroleum diesel. The apparatus are conical flask, magnetic stirrer regulator hotplate, muffle furnace, separating funnel, pipette and burette, vacuum evaporator, GC-MS and FTIR instruments, fisher brand hydrometer, automated Pensky-Martens closed cup, bomb calorimeter, thermometer, refrigerator, copper strip and viscometer Figure 2 is the pictorial representation of propan-2-ol, methanol, sulphuric acid (H_2SO_4), potassium hydroxide (KOH) and phenolphthalein indicator respectively.

Figure 2: Pictorial representation of some biodiesel production materials

3.1.1 Material Preparation

The sample used in this work is called neem oil from neem seeds (*Azadirachta Indica)*, it is purchased from a local vendor in Kano. The eggshells were sourced from restaurants, tea and indomie joints as waste and any other aforementioned material were bought from the market. Figure 3(a) & (b) portrayed the neem oil and eggshells respectively.

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(a) Neem oil sample **(b)** eggshells

Figure 3: Neem oil Sample and Eggshells

3.1.2 Neem oil preparation

- Determination of Neem oil Acid Value

25 ml propan-2-ol was added to 1.14 g of sample. It was dissolved by shaking the mixture, and then three droplets of phenolphthalein were added up to the solution. The mixture was titrated against 0.1 M KOH to pink colour. The titer value was found to be 6.1ml. The acid value and the percentage acid were calculated as thus;

$$Acid\ value = \frac{Titre\ Value\ (ml) \times Normality\ of\ KOH \times Molar\ mass\ of\ KOH}{mass\ of\ sample\ (g)} \quad (1)$$

$$\%FFA = \frac{Acid\ Value}{2} \quad (2)$$

The acid value and percentage free fatty acid values were found to be not within the acceptable range and hence the oil will undergo esterification reaction process.

- Esterification reaction

1000g of the oil was weighed and poured into a conical flask; the conical flask was sealed and heated to about 60 ºC with continuous stirring using a magnetic stirrer regulator hotplate. The quantities of alcohol (i.e. methanol) and sulphuric acid required for esterification were calculated as;

$$M_{methanol} = 2.25 \times \%FFA \times mass\ of\ oil \quad (3)$$

$$M_{H_2SO_4} = 0.05 \times \%FFA \times mass\ of\ oil \quad (4)$$

Where, $M_{methanol}$ is mass of alcohol (methanol) and $M_{H_2SO_4}$ is mass of sulfuric acid. The mass of methanol and that of sulfuric acid required were found to be 337.5g and 7.5g respectively. 7.5g of Sulphuric acid (H_2SO_4) was used as catalyst, and 337.5g of methanol was mixed with the catalyst and it was added to the sample at 60 ºC and then heated together for 1h between the temperatures of 60 and 65 ºC with continuous stirring on the magnetic stirrer regulator hot plate. The esterified oil was transferred and remained into a separating funnel for 60 minutes. The oil was collected at the lower part of the funnel and the acid/water solution on top was discarded.

After successful esterification, titration was carried out again and the FFA was determined to be 0.47 which is less than 0.5. This implies that the sulfuric acid modified the free fatty acid molecules where the methanol attached to them and hence became prepared for biodiesel production, this is in line with (Kaisan et al., 2014).

3.1.3 Catalyst Preparation

The eggshells were cleaned and washed with clean water for the removal of the thin layer. After which, they were dried under the sun for 2 days and thereafter placed in a metal container heated for 3hrs using biomass and finally thermally calcined in a muffle furnace at 900°C for 3hrs. After the calcinations process, the $CaCO_3$ in the eggshell was transformed to CaO as catalyst. This is in accordance with (Kavitha et al., 2019).

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a. Before **b.** After

Figure 4**:** Before and after calcination inside the furnace

3.2 Biodiesel Production

The prepared Neem oil undergoes transesterification process for the production of the biodiesel as below. A given concentration of 0.5% (wt of oil) of CaO was added to 27% (wt of 0il) of methanol, stirred and it was dissolved completely. 35ml of the prepared oil was poured in a flask and the CaO-methanol mixture was added to it. The solution was agitated for 70 minutes at 65^0C using magnetic stirrer regulator hot plate and then poured into a separating funnel and remained there for 24 hours. After 24hrs, two layers were produced, biodiesel and glycerin as shown in Figure 5. The glycerol is at the bottom and the top layer is biodiesel. This is common with fatty acids such as oleic acid. The glycerol was drained through the nozzle at the bottom of the flask, leaving the biodiesel behind. This is in line with the procedure of (Kaisan et al., 2013). The biodiesel was washed for the removal of the soluble components. Hot water was sprayed on top of the biodiesel. After settling down, the biodiesel was collected and it was dried with the aid of vacuum evaporator. After the drying, the pure product became clear. This is in accordance with the procedure of (Banik et al., 2018). The transesterification of the biodiesel was carried out nine (9) times at constant temperature and volume of methanol as mentioned above. Catalyst concentration were varied three (3) times 0.5%, 1.0% and 1.5% (wt of oil) and for each catalyst concentration the reaction time were also varied three (3) times 70min, 110min and 150min as illustrated in table 2. The nine (9) samples produced are shown in Figure 6.

Figure 5: Pictorial representation of biodiesel separation from glycerin

Table 2. Variation of parameters in the production of neem oil biodiesel using calcined eggshells catalyst

Fixed Parameters		Variation Parameters	
S/N	**Catalyst concentration (% (wt of oil)):**	**S/N**	**Reaction time (min):**
1	0.5	i	70
		ii	110
		iii	150
2	1.0	i	70

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Fixed Parameters		Variation Parameters	
S/N	**Catalyst concentration (% (wt of oil)):**	**S/N**	**Reaction time (min):**
		ii	110
		iii	150
3	1.5	i	70
		ii	110
		iii	150

Figure 6: Nine (9) samples of biodiesel produced

3.2.1 Blending

Petroleum diesel was taken as the only fuel constituent for the blending of the biodiesel. The biodiesel-diesel blend was carried-out in the ratio 10:90, 20:80 and 30:70 by volume respectively. Whereas the biodiesel and the conventional diesel were reserved for final comparison on the performance and emission characteristics parameters. B10, B20 and B30 were the denotations for the blends and the denotation for biodiesel alone and that of pure petroleum diesel alone were B100 and B0 respectively. This is in accordance with (Sani et al., 2018).

3.3 Characterization Analyses

3.3.1 Gas Chromatography – Mass Spectrometry (GC-MS) Analyses

GC-MS analysis was carried out and the biodiesel (methyl ester) percentage present in B100 was determined. The contents of FAMEs in the neem biodiesels was also analysed by the used of GC-MS. Separation was accomplished using a capillary column DB-5MS (30m×0.32 mm, 0.25 µm film thickness). The gas used as carrier was helium, which had a flow rate of 1.5 mL/min. The column temperature was set to increase from 120°C to 300°C at a speed of 10°C per minute, this is in accordance to Kaisan et al. (2018). The temperature of both detector and injector were adjusted to 200°C. A rip mode was used in the injection of 0.1L volume of methyl ester sample using a split ratio of 1:10. The mass spectrometer in conjunction with electron impact mode of ionization was adjusted and scanned in the range of m/z 50–550, this is also in accordance with (Kaisan et al., 2018). GC-MS analysis was used as a medium that studied the chemical contents of the produced biodiesel. The total ion chromatogram is responsible for the display of the major peaks. Based on the library match software (NO. NIST, 02), the peak corresponds to a FAME content of the biodiesels. By running the standards under similar experimental conditions and relating the respective retention time data which were validated by MS analysis, the identified FAMEs were confirmed. The process were based on the following conditions: column oven temperature of 60°C; injection temperature of 200°C; injection pressure of 112.8 kPa, this is in accordance to (Xingzhong et al., 2008).

3.3.2 Fourier-Transformed Infrared Spectroscopy (FTIR) Analyses

In FTIR, the neem biodiesel sample was examined using 25x2mm sodium chloride (NaCl) polished discs. By scanning the two clean discs placed in the instrument together, the background spectra were acquired. A drop of the respective sample of the neem biodiesel was set on one of the NaCl disc and covered

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by the other one. In that form, they were inserted in a Perkin-Elmer Spectrum Carry 630FTIR which were scanned on the range of 4000-650 cm^{-1}, this is in line with the work of (Tariq et al., 2011).

3.4 Determination of Physico-chemical Properties of the Biodiesel

The physicochemical properties of the biodiesel evaluated includes; Yield, density, viscosity, flash point, cloud point, cetane number, acid value, iodine value, saponification value and calorific value. The experiments were conducted in line with the American Society of Testing and Materials (ASTM) D6751 Standards in Chemical engineering department laboratory of ABU Zaria. Thus, in this study, the quality of biodiesel from neem seed oil with the use of calcined eggshell catalyst was evaluated with regard to the physico-chemical properties mentioned earlier in this paragraph which were related to ASTM standard for justification.

3.5 Engine Performance and Emission Test

The engine test was carried out in a stationary single-cylinder compression ignition engine (P8750). The engine test was conducted and the following parameters where evaluated; brake power, brake thermal efficiency, brake specific fuel consumption and brake mean effective pressure after running the engine on biodiesel, diesel and biodiesel-diesel blends. The computer that is in connection with the engine during the experiment have an installed software which recorded the exhaust and inlet temperatures, fuel consumption, air flow rate, load and speed. The engine was programmed to run at constant speed of 1500 rpm under constant load of 4.5N. Gas analyser which is having a sensor cable that was inserted in the immediate outage of the exhaust pipe of the engne and a screen that display the emission parameters was also used for the determination of the exhaust gas emissions. The test was in line with the procedure used by (Sani et al., 2018).

4. RESULT AND DISCUSSION

4.1 Biodiesel Production/Optimization Result

In Figure 7, a biodiesel yield of 94% was obtained using 0.5% (wt. of oil) calcined eggshell catalyst at both 70 and 110 minutes of reaction time. At a catalyst concentration of 1.0% (wt. of oil), the yield increased to 100% at 70 minutes. Conversely, using 1.5% (wt. of oil) catalyst produced approximately 83% yield at the same reaction time. Therefore, the optimum biodiesel yield of 100% was achieved with 1.0% (wt. of oil) calcined eggshell catalyst at a reaction time of 70 minutes.

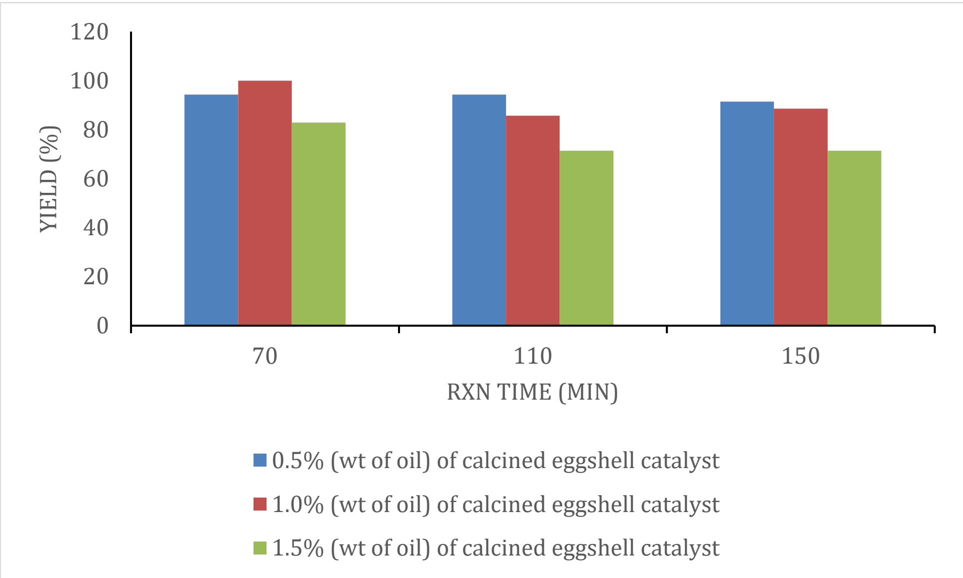

Figure 7: Biodiesel production result by varying catalyst at different reaction time

4.2 GC-MS Analysis Results

The percentage of the methyl ester present in the optimum biodiesel produced from neem oil with the used of calcined eggshell catalyst was evaluated using Gas Chromatography - Mass Spectroscopy (GC-MS) analysis of the biodiesel as presented in Figure 8.

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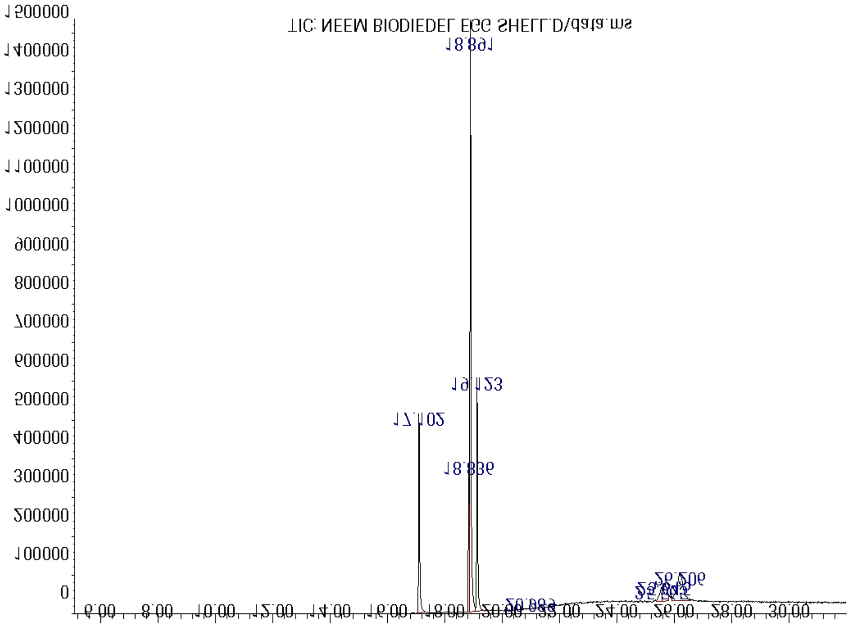

Figure 8: GC-MS analysis of biodiesel

The graph shows eight vibrational peaks which means, eight distinct compounds are contained in the sample. It is clearly portrayed from graph that peaks are proportional to abundance which demonstrate that lower intensity peaks indicate compounds with lower abundance and vice vasa. The methyl ester compounds were determined by GC-MS data library matching pattern which display the corresponding compound of the peaks as shown in Table 3.

The main methyl esters in the Neem biodiesel are hexadecanoic acid methyl ester ($C_{17}H_{34}O_2$), 9,12-octadecadienoic (Z,Z) acid methyl ester ($C_{19}H_{34}O_2$), 9-Octadecenoic acid methyl ester (E)- ($C_{19}H_{36}O_2$) and Methyl stearate $C_{19}H_{38}O_2$. Their respective percentages are 13.57 %, 8.16 %, 42.83 % and 15.43 %. Some other esters discovered in the biodiesel are Eicosanoic acid methyl ester ($C_{21}H_{42}O_2$) 0.99 %. The profile disclosed that, 9-octadecenoic acid methyl ester, (E)- ($C_{19}H_{36}O_2$) is the overriding compound in the solution with 42.83 %.

The profile of the fatty acid methyl ester is the most important parameter that measure out the propensity or aversion of any raw material to be use in biodiesel production (Kaisan et al., 2018). The outcome in Figure 8 show that, the monounsaturated 9-octadecenoic acid methyl ester, (E)- is the mass excessive ester in the optimum Neem biodiesel. It is an advantageous compound with the spirit that guarantee the reliability of the biodiesel, this is because, higher intensity of unsaturation in the methyl ester fatty acid clamp down its convenience for use as a fuel owing to high polymerisation tendency which is caused by strong oxidizing agent.

As shown in Figure 8, the results obtained from the GC–MS analysis, methyl esters account for 80.98 % of the total composition, as clearly shown in the output. The products are predominantly methyl ester compounds, which is confirmed by the GC–MS results, indicating that biodiesel constituents constitute the major fraction of the analyzed compounds.

Table 3. Determination of peaks proportionates to fatty acids chain numbered 1-8

Peak No	Retention time (mins)	Area(%)	Fatty Acids	Chemical formulae
1	17.102	13.57	Hexadecanoic acid	$C_{17}H_{34}O_2$
2	18.836	8.16	9,12-Octadecadienoic acid (Z,Z)	$C_{19}H_{34}O_2$
3	18.891	42.83	9-Octadecenoic acid	$C_{19}H_{36}O_2$
4	19.123	15.43	Methyl stearate	$C_{19}H_{38}O_2$

Peak No	Retention time (mins)	Area(%)	Fatty Acids	Chemical formulae
5	20.989	0.99	Eicosanoic acid	$C_{40}H_{80}O_4$
6	25.565	2.42	1-Naphthamide	$C_{11}H_9NO$
7	25.645	4.95	Sarcosine	$C_3H_7NO_2$
8	26.206	11.66	Fumaric acid	$C_4H_4O_4$

At higher temperatures in compression ignition engines, the oxidizing process can be speedy causing polymerization of fatty acid methyl esters which may subsequently cause rapid gumming of the engine (Gopinath *et al.*, 2010). Meanwhile, raw materials with high proportion of poly unsaturated acid is unsuitable for use in biodiesel production. Though the prevalent fatty acid methyl esters for Neem biodiesel are hexadecanoic acid methyl ester, 9,12-octadecadienoic (Z,Z) acid, methyl ester, 9-Octadecenoic acid, methyl ester (E)-, and Methyl stearate are mono-unsaturated, their relation with oxygen might be insignificant to cause rapid peroxidation. These prevalent esters are in line with the findings of Kaisan, e al. (2018) on Neem and Jatropha biodiesel using NaOH catalyst whose results gave (54.69%, 3.88% and 10.64%) and (53.44%, 17.77% and 14.25%) for methyl octadecanoate, methyl hexadecanoate and methyl pentadecanoate respectively (Kaisan et al., 2018). Neem and Jatropha oil methyl esters have made known to be reliable raw materials for biodiesel production (Kaisan et al., 2018). The prevalent esters obtained from them are methyl octadecanoate and methyl hexadecanoate like those obtained in the Neem biodiesel using calcined eggshells catalyst. hence, it could be concluded that Neem biodiesels using calcined eggshell catalysts is combustible, renewable, sustainable, reliable and suitable for use in CI engines.

4.3 **FTIR Results**

The FTIR spectrum of the biodiesel produced from Neem seed oil using calcined eggshell catalyst is presented in Figure 9. The spectrum confirms the successful transesterification of the oil into fatty acid methyl esters (FAMEs). A prominent absorption band was observed at 1744 cm^{-1}, corresponding to the C=O stretching vibration of ester carbonyl groups, which is a key indicator of biodiesel formation. This peak verifies the conversion of triglycerides present in the raw Neem oil into methyl esters.

Strong absorption bands at 2922 cm^{-1} and 2855 cm^{-1} represent the asymmetric and symmetric stretching vibrations of aliphatic $-CH_2$ and $-CH_3$ groups, characteristic of long-chain fatty acid alkyl structures. The peak at 3008 cm^{-1} corresponds to the =C–H stretching of unsaturated alkenes, indicating the presence of unsaturated fatty ester chains commonly found in vegetable-oil-derived biodiesel.

Additional ester-related peaks appeared between 1244 and 1100 cm^{-1}, representing C–O and C–O–C stretching vibrations of the ester linkage. The absorption bands at 1461–1438 cm^{-1} correspond to the bending vibrations of CH_2 groups, while the peak at 1360 cm^{-1} signifies symmetric bending of CH_3 groups. Smaller peaks in the region of 1017–879 cm^{-1} represent out-of-plane bending and additional C–O–C vibrations associated with methyl esters.

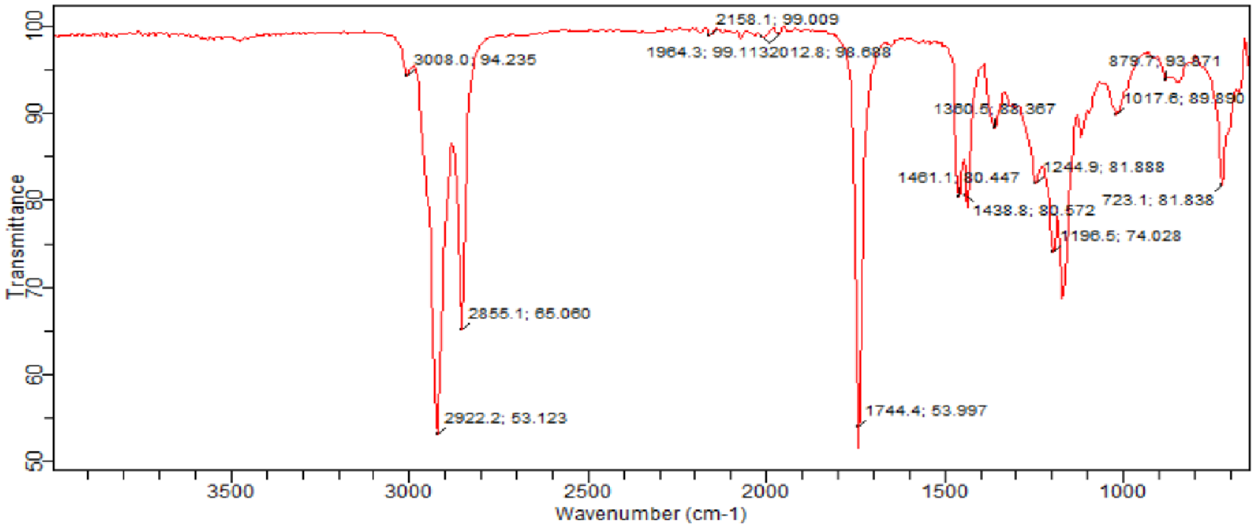

Figure 9: FTIR spectrum of biodiesel

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Summary of all major peaks and their functional group assignments were displayed in Table 4. Collectively, the presence of the ester carbonyl band, C–O stretching peaks, and aliphatic hydrocarbon vibrations confirms that the product obtained is biodiesel. The FTIR spectrum thus validates the effectiveness of the calcined eggshell catalyst in converting Neem seed oil into high-quality fatty acid methyl esters.

Table 4. Functional groups present in Neem biodiesel

Wavenumber (cm^{-1})	**Functional group/Vibrations**	**Interpretation**
~3008	=C-H Stretching (alkenes)	Unsaturation in fatty acid chains
2922	Asymmetric $–CH_2$ Stretching	Long-chain hydrocarbons
2855	Symmetric $–CH_3$ stretching	Aliphatic groups in FAME
1744	C=O stretching (ester carbonyl)	Strong confirmation of biodiesel formation
1461, 1438	CH_2 bending (scissoring)	Methylenes in fatty acid esters
1360	CH_3 symmetric bending	Methyl groups in esters
1244, 1170	C–O stretching (ester bond)	Characteristic of methyl esters
1100-1017	C–O–C stretching	Ester linkage vibrations
879-723	Out-of-plane bending / C–O–C	Additional ester-related vibrations

4.4 Physicochemical properties Result

The results show that the NSO biodiesel yield is 100% and that the production of the biodiesel from NSO through transesterification process bring about a considerable change in the initial NSO physical and thermal properties. The NSO properties is from the findings of Djibril et-al, 2015. The comparative analysis of the NSOB properties with those of the NSO communicates a decrease of the viscosity (49.79 to 2.1 $mm^2.s^{-1}$), of the density (912 to 880.1kgm^{-3}), of the acid value (9.10 to 0.47mgKOH/g of oil), of the iodine value (74.82 to 18.53 gI_2/100g of oil), of the saponification value (200.54 to 119.212$mg.g^{-1}$), of the flash point (227 to 137°C) and it also communicate an increase of the gross calorific value (39.53 to 43.21 $MJ.kg^{-1}$). The NSOB properties are equally compared with standard as shown in Table 4. It can be regarded as an alternative fuel for CI engines. The physical and thermal properties obtained from the NSOB in this study are also similar to those of diesel.

Table 5. Physico-chemical Properties of Neem oil biodiesel compared with standard

Parameters	This study	Standard limit: min.	max.	Test standard
Yield(%)	100	96.5		EN 14103
Density(kgm^{-3})	880.1	880		ISO17828
Viscosity(mm^2/s)	2.1	1.9	6.0	ASTM D6079
Flash point(°C)	137	130.0		ASTM D93
Cloud point	7	-3	12	ASTM
Cetane number	56	47		ASTM D613
Acid value(mgKOH/g of oil)	0.47		0.5	ASTM D664
Iodine value(gI_2/100g of oil)	18.5274		120	EN 14214
Saponification value($mg.g^{-1}$)	119.212	-		
Calorific value ($MJ.kg^{-1}$)	43.21	42	46	ASTM

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4.5 Engine Performance Results

4.5.1 Brake Thermal Efficiency

The result shows that, brake thermal efficiency is increasing with an increase of the biodiesel in the biodiesel diesel blend at constant torque and speed of 4.5Nm and 1500rpm respectively. The smaller value of brake thermal efficiency is found in B10 with an increase in B20, subsequent increase in B30 and greater value is found in B100 whereas the pure conventional diesel B0 has the least value; this is in line with the findings of (Sani et al., 2018). The values of brake thermal efficiency are presented in Figure 10.

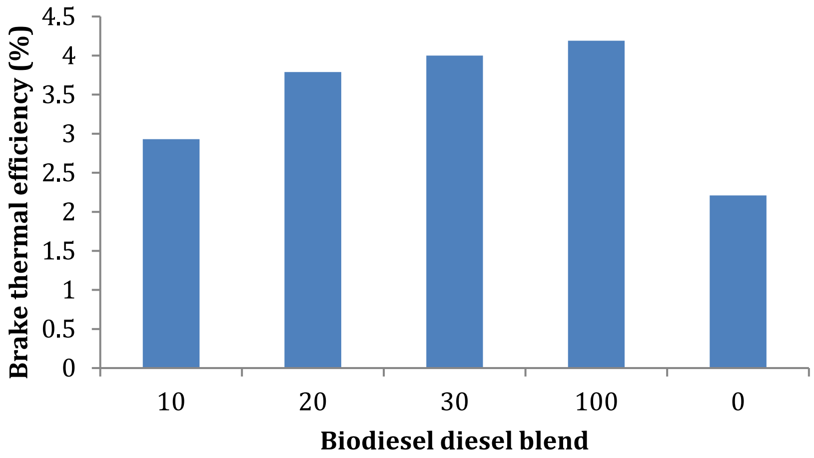

Figure 10: Brake thermal efficiency (BTE)

4.5.2 Exhaust Gas Temperature

According to Atmanli and Yilmaz (2020), the exhaust gas temperature (EGT) manifest the heat generated within the combustion chamber and has sufficient influence on the formation of pollutants that have negative effects in both human and environment. The variation in EGT (from a stationary single cylinder compression ignition engine) as a function of biodiesel blend ratios is given in Figure 11. This Figure presents the values of exhaust gas temperature for B10, B20, B30, B100 and B0 at constant torque and speed. As in brake thermal efficiency, the exhaust gas temperature is increasing with increase in biodiesel in the biodiesel-diesel blend. B10 has the lowest value as 237^{o}C and B100 with the highest value as 294^{o}C whereas B0 (diesel) has the least value as 201^{o}C. As reported by Hellier *et al.* (2015), rates of soot oxidation are directly proportional to temperatures, the reason is that, the in-cylinder temperatures are soot exhaust emission dependent. Hence, the in-cylinder temperatures are also directly proportional to exhaust temperatures.

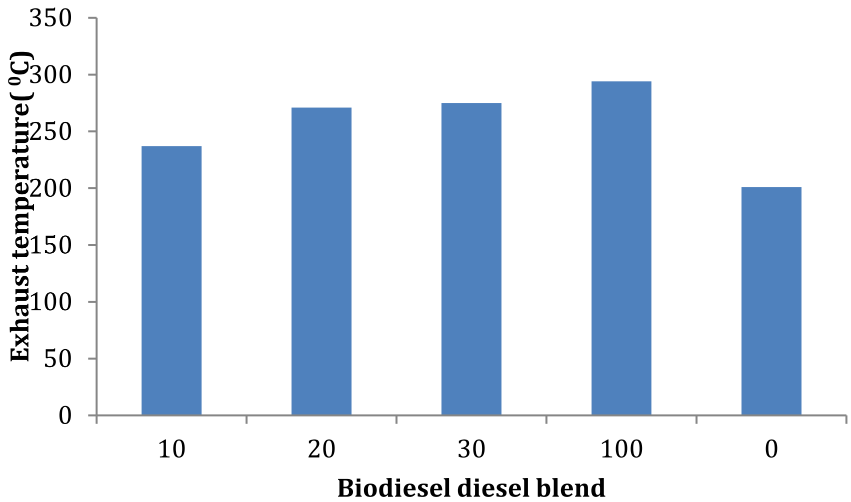

Figure 11: Exhaust gas temperature

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4.5.3 Brake Power (Bp)

The brake power values portrayed in Figure 12 were obtain from single cylinder stationary engine test. B10 (10% biodiesel and 90% diesel) has the minimum value of brake power, it is observed that the brake power increases while the percentage of biodiesel in the blend increases, therefore brake power increase in B20 (20% biodiesel and 80% diesel) and subsequent increase in B30 (30% biodiesel and 70% diesel) were noted and B100 (100% biodiesel and 0% diesel) has the maximum value of brake power in kilo watts whereas B0 (0% biodiesel and 100% diesel) has the least value of Bp.

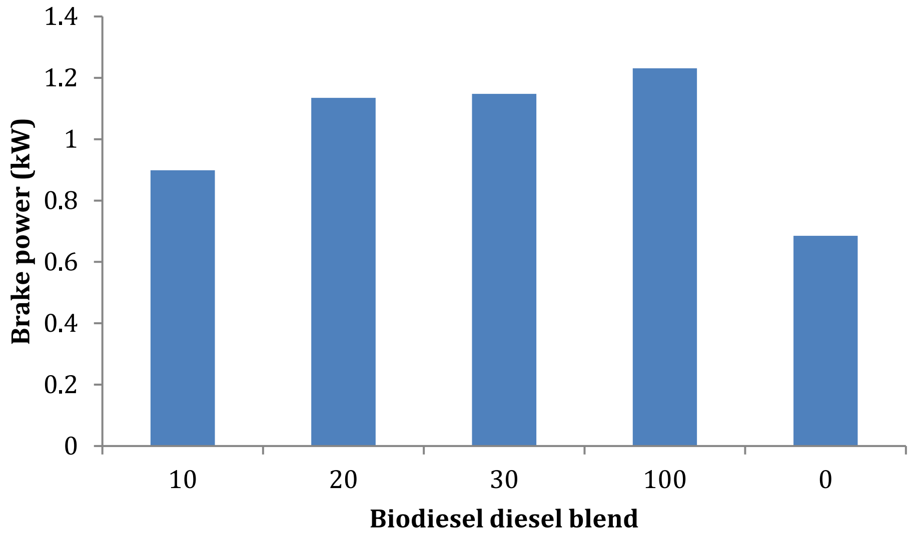

Figure 12: Brake Power (BP)

4.6 Emissions result

4.6.1 UHC Emission

Figure 13 illustrates the variation of unburnt hydrocarbon emissions at constant load and speed based on biodiesel blend ratios. The lowest hydrocarbon emissions were resulted from the burning of B20 and B30 in a stationary single cylinder CI engine, under the same usage process B10 and B100 give the highest UHC emissions as portrayed in same Figure 13. The higher level of UHC emissions is as a result of incomplete combustion of fuel-air mixture.

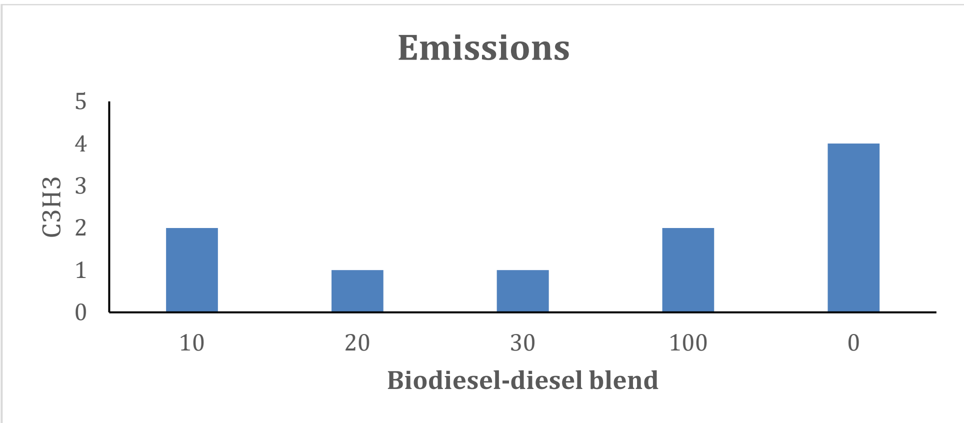

Figure 13: Unburnt hydrocarbon (UHC) emissions

4.6.2 NOx Emission

NOx is also an important emission parameter which is formed by the combination of nitrogen and oxygen inside the combustion chamber. The three key elements fostering the development of NOx emissions were high temperature, oxygen handiness, and time covered during combustion (Prabhu et al., 2021). Figure 14 depicts the generation of NOx for biodiesel-diesel blend ratios. The result shows that B30 has the lowest NOx emissions and all other biodiesel-diesel blends are in a similar manner having NOx emissions below that of the pure diesel, which may be connected with the oxygen content and the level of unsaturation of the fatty

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acid, which may provide low temperature for NOx emissions, similar case was experienced by (Atmanli & Yilmaz, 2020).

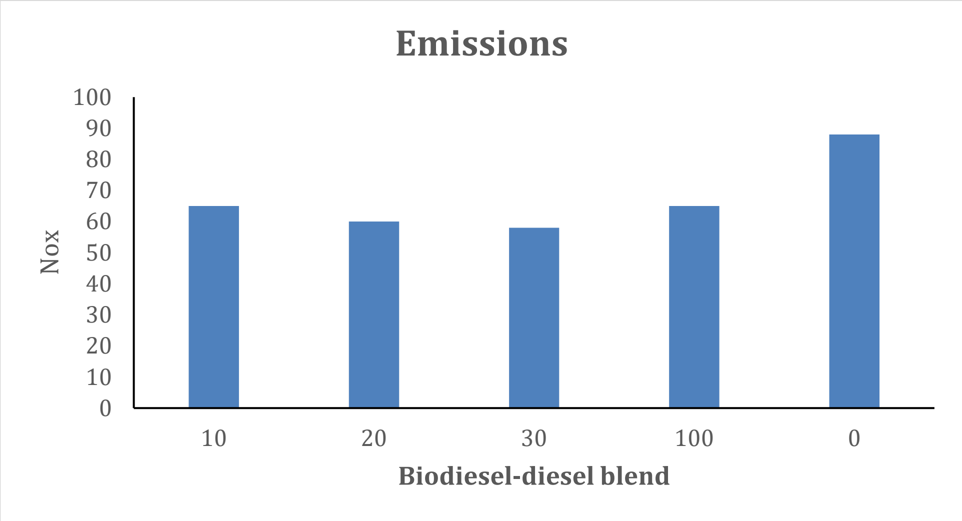

Figure 14: Nitric oxides (NOx) emissions

4.6.3 CO Emissions

Similar to HC emission, incomplete combustion is also a source of CO emission. Fuel rich zones are carefully weighed to be the prima parameter that causes CO emission, as this emission is realised to be responsive to air fuel ratio (Ashok et al., 2019). The diesel CO emission is determined to be more dangerous than that of biodiesel. As the essential characteristics of biodiesel that is an oxygenated fuel while on the other hand diesel is a mineral oil made up of pure hydrocarbon chain. Figure 15 was aimed to show the variation of carbon monoxide at constant load of 4.5N and constant speed of 1500rpm but it can be observed that no carbon monoxide emissions was recorded, this could be that more air was supplied into the CI engine and hence enough oxygen was readily available in the combustion chamber for complete combustion of the various biodiesel and their blends as reported by (Abed et al., 2019).

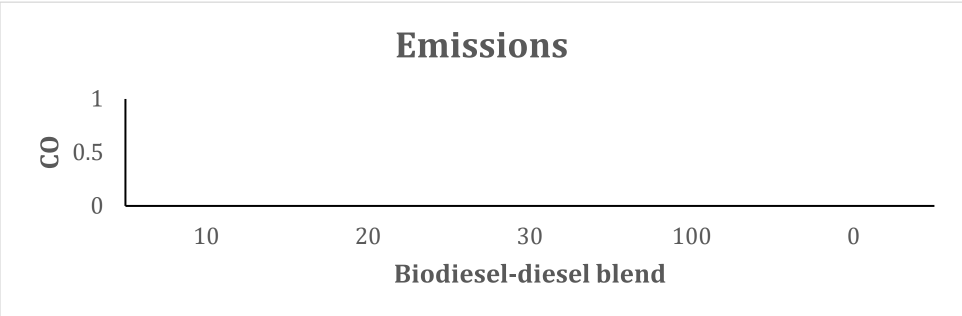

Figure 15: Carbon monoxide (CO) emissions

4.6.4 CO_2 Emissions

Carbon dioxide emissions may be attributed to the availability of additional oxygen molecule within the fuel that helps to promote complete combustion of fuel supplied to the engine. When there is availability of enough oxygen, CO will be converted to CO_2 by the hydroxyl radicals formed during the combustion process. Figure 16 shows that B30 has the lowest CO_2, B20 and B30 have CO_2 emissions equivalent to that of the pure diesel while B100 has CO_2 emissions higher than that of all the blends including the pure diesel.

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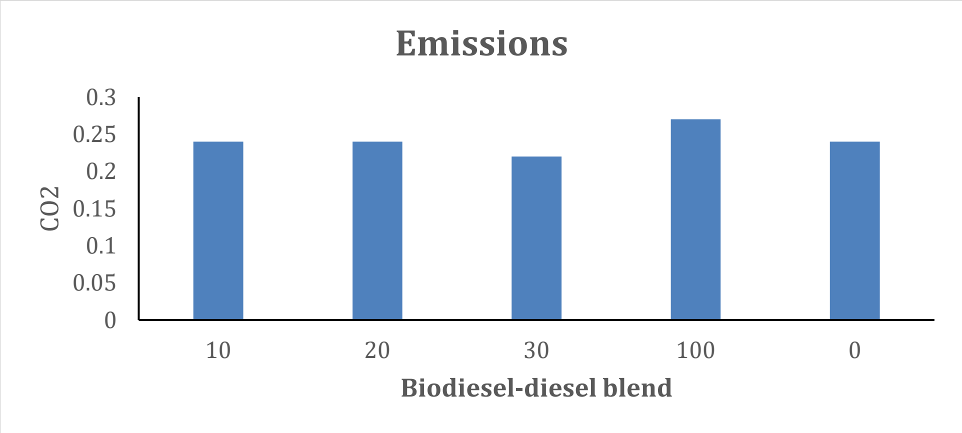

Figure 16: Carbon dioxide (CO_2) emissions

5. Conclusion

This study demonstrated that calcined eggshell-derived CaO is an effective and economically viable catalyst for biodiesel production from Neem seed oil. Among the nine biodiesel samples produced, the optimum yield of 100% was achieved using 1.0% (wt of oil) catalyst at 70 minutes reaction time. The resulting biodiesel exhibited favourable physico-chemical properties, contained 80.98% methyl esters, and showed clear ester-related functional groups in the FTIR spectrum, confirming successful transesterification.

Engine tests revealed that the optimum biodiesel and its blends performed efficiently in a CI engine. B30 and B100 produced the highest brake thermal efficiency and brake power, while B10 recorded the lowest exhaust gas temperature. Emission results further showed reduced UHC, NOx, and CO_2, with zero CO across all blends.

Overall, the produced biodiesel is renewable, environmentally friendly, and suitable for CI engine applications, demonstrating strong potential as a sustainable alternative to conventional diesel.

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