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Original Article

Biodiesel Production and Characterization for Croton Oil Methyl Ester and Its Blends with Graphene and Graphene Oxide Nanoparticles

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***Abstract** - The production of biodiesel from non-edible oil feedstock is gaining attention around the world as a sustainable alternative to fossil diesel that poses no threat to food security. Hence providing motivation to explore new and potential sources. Croton oil was used to make biodiesel in this study through the transesterification process, and the viscosity, density, calorific value, and flash point are some of the properties tested for comparison with corresponding properties of fossil diesel. Properties of diesel-biodiesel blends and blends with nanoparticles (Graphene and Graphene Oxide) were tested to establish the effect of nanoparticles on biodiesel properties. The best transesterification conditions for biodiesel production were found to be Croton oil to methanol ratio of 1:1, stirring frequency of 310 Hz, temperature of 60-65°C, and reaction time of 1 hour. The physicochemical properties of the Croton methyl ester were found to lie within the range of existing biodiesel standards. Fourier Transform Infra-red (FTIR) analysis revealed comparable spectra for Croton methyl ester and diesel. The addition of graphene and graphene oxide nanoparticles in proportions of 25 ppm, 50 ppm, and 100 ppm showed a marginal effect on both the density and viscosity of the fuel blends and a slight increase in the calorific fuel value.*

***Keywords** - Biodiesel, Diesel, Fourier Transform Infra-red (FTIR), Nanoparticles, Transesterification*

I. INTRODUCTION

Globalization and population growth have resulted in increased consumption of petroleum products around the world, which has a detrimental influence on the environment and creates a variety of health issues as a result of hazardous emissions. Due to the depletion of petroleum resources, a concentrated effort has been made to develop environmentally safe and sustainable alternative energy sources to meet the expanding energy demand [1]. Biodiesel, a diesel engine alternative fuel, is one of the promising renewable energy sources. Biodiesel is made up of long-chain fatty acid esters that are generated from plant oil or animal fat[2]. Vegetable oils should not be used directly in diesel engines due to their high viscosity. [3].

Direct use, blending, micro-emulsion, pyrolysis, and transesterification are the methods for converting vegetable oil to biodiesel [4]. Transesterification is the most widely utilized procedure because it is easier, more effective, and less expensive than the other methods. The esterification process is performed to lower the acid value If it is higher than the recommended value.

Various biodiesel sources have been examined, including algal oil with high free fatty acid concentration [5]; several researchers have considered Croton megalocarpus, which is available in Kenya, as a promising biodiesel source. [6], [7].

The trend is to lower the viscosity, increase the energy content, reduce NOx emissions [8]to meet the latest emission standard (Euro 6 emissions standards). In recent decades, several researchers have attempted to address the biodiesel challenges through different approaches. Some of the studies reported in the literature have shown that the addition of certain nanoparticles to biodiesel improves engine combustion and performance characteristics and reduces emissions. However, there is limited literature on the use of nanoparticles with biodiesel from some potential feedstocks such as Croton and Oleander oils. Additionally, the effect of the nanoparticles on the physicochemical composition and properties has not been extensively studied.

Quality control of biodiesel has drawn significant research attention, especially regarding viscosity requirements and biodiesel stability under long-term storage. The determination of the chemical composition and physical properties of biodiesel is one of the most significant aspects of biodiesel quality analysis.

In order to determine free fatty acid in fish oils for biodiesel generation, Aryee et al. [9] used a Fourier transform infrared (FTIR) spectroscopy approach. The method was found to be a versatile and practical alternative for determining the FFA content of crude for biofuel production. Oliveira et al. [10] developed calibration models based on FTNIR (transflectance-fiber optics), and FTIR-ATR combined with PLS and ANN analysis and showed that the model might be used to determine the methyl ester levels in biodiesel blends. Furthermore, Rosset and Perez-Lopez [11] revealed that FTIR and Gas Chromatography (GC) might be utilized to monitor heterogeneous catalyst biodiesel generation.

In order to fill the gap evident in literature, it is necessary to prepare biodiesel from raw oil and conduct comprehensive property analysis to establish the effect of nanoparticles on physicochemical properties of biodiesel from new potential sources, such as Croton oil. Furthermore, most previous research was focused on the effect of nanoparticles on the combustion and emission characteristics of diesel and biodiesel-fuelled engines, with little attention paid to their impact fuel qualities. Hence the need for comprehensive property analysis to ascertain the influence of nanoparticles.

The goal of this research was to make biodiesel from Croton oil via transesterification and evaluate the effects of graphene and graphene oxide nanoparticles on the biodiesel's physicochemical properties.

II. MATERIALS AND METHODS

A. Materials

Croton oil was supplied from Eco Fuel Company in Nanyuki, Kenya, for this investigation. The properties of the Croton oil, methanol, and sodium hydroxide used in the transesterification process are presented in Table 1, while the properties of the nanoparticles are presented in Table 2.

Table 1. Properties of materials used in transesterification

Material	Property	Specification
Croton oil	Appearance	Viscous amber liquid
	Boiling Point	> 230 °C
	Pour Point	-20 °C
	Viscosity a 40°C	30.4 mm^2/s
	Specific Gravity at 20°C	0.92
	Solubility in Water	Not soluble; not miscible
	Flashpoint	250°C
	Flammability Limits	Not Available
	Vapor Pressure	Not Available
Methanol: analytical grade	Purity	>99%
Sodium hydroxide	Purity	98%

Table 2. Graphene and Graphene oxide properties

	Graphene	Graphene oxide
Company	Nanoshel	Adnano Technologies
Color	Black	Black
Morphology	Flaky	Fluffy, Very Light Powder
Layer flake	4-6 layers (50-80%)	1-3 layers
Purity	99.5%	~99%
Thickness	2-4nm	~0.8-2 nm
Lateral size	~5 µm (±3%)	~5-10 µm
Bulk density	~0.10 g/cm3	0.121 g/cm3
Thermal conductivity	3000W/mK	-
Carbon Content	>99%	~60–80%
Oxygen Content	-	~15-32%

B. Methods

a) Biodiesel production

Transesterification, a chemical reaction involving Croton oil and short-chain alcohol (methanol) in the presence of sodium hydroxide catalyst, was used to make biodiesel from Croton oil. Fig. 1 describes the biodiesel production process.

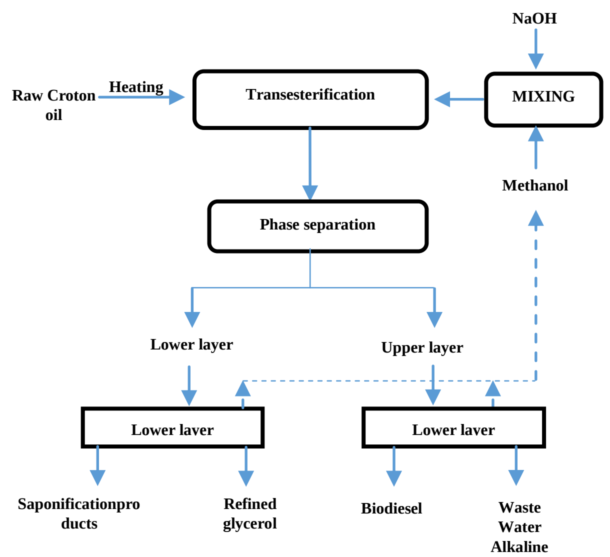

Fig. 1 Schematic process of biodiesel production through transesterification [12]

One percent weight/volume of sodium hydroxide (1% w/v of NaOH) was dissolved in methanol and mixed with Croton oil. The mixture was heated for one (01) hour at a steady temperature of 60°C. The mixture was separated by transferring the solution into a separating funnel and allowing it to settle for 1-2 hours. A top ester layer and bottom glycerol layer were formed. The ester was drained and washed with warm distilled water until the wash water was clear. It was then dried in an oven at a constant temperature of 118°C to obtain Croton Oil Methyl Ester (CROME).

Fig. 2 Steps in biodiesel production through transesterification

b) Fuel Blends Preparation

Different fuel blend samples were prepared and tested, and their physicochemical properties were compared with diesel. From the literature, the best performance of biodiesel blend with diesel fuel is in the range of 5 to 20% biodiesel in blend with diesel [13][14]. A 20% biodiesel-diesel blend was used with nanoparticle concentrations of 25 ppm, 50 ppm, and 100 ppm, as stated in Table 3.

Table 3. Fuel samples tested

No.	Fuel sample	Description
1	D100	100% diesel
2	B100	100% CROME
3	B20	20% CROME + 80% diesel
4	B20+G25 ppm	20% CROME + 80% diesel + 25 ppm of graphene
5	B20+G50 ppm	20% CROME + 80% diesel + 50 ppm of graphene
6	B20+G100 ppm	20% CROME + 80% diesel + 100 ppm of graphene
7	B20+GO25 ppm	20% CROME + 80% diesel + 25 ppm of graphene oxide
8	B20+GO50ppm	20% CROME + 80% diesel + 50 ppm of graphene oxide
9	B20+GO100 ppm	20% CROME + 80% diesel + 100 ppm of graphene oxide

Fig. 3: shows the materials used to prepare the nano-fuel, while Fig. 4 shows the various samples tested.

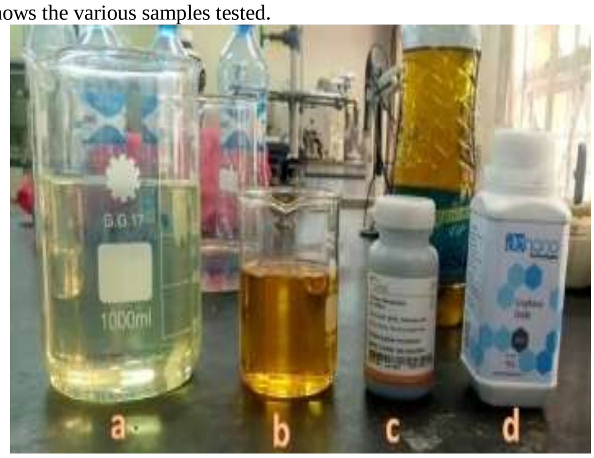

Fig. 3 Materials (a) Diesel; (b) CROME; (c) Graphene; (d) Graphene oxide

Fig. 4 Test samples (a) B20; (b) B20 + G25 ppm; (c) B20 + G50 ppm, (d) B20 + G100 ppm; (e) B20 + G025 ppm; (f) B20+G050 ppm; (g) B20 + G0100ppm

The blends were prepared by measuring 800 ml of diesel and 200 ml of CROME using a measuring cylinder and mixing the two fuels in a 2000 ml beaker. The mass of nanoparticles required was measured using a sensitive mass balance ENTRIS224-1S, with a sensitivity of 0.0001 g. The fuel mixture with nanoparticles was then subjected to mechanical stirring at 200 rpm using a magnetic stirrer N2400-3010 for 5 minutes to ensure the uniform distribution of nanoparticles in the fuel sample.

c) Chemical Composition Analysis and Test

The chemical composition of diesel, raw Croton oil, Croton methyl ester (B100), and its blends with Graphene and Graphene oxide were investigated through Fourier Transform Infra-red (FT-IR) analysis. All of the samples were also subjected to physicochemical property testing such as density, viscosity, calorific value, and flashpoint.

The list of equipment and devices used in the physicochemical property analysis of the fuel samples is shown in Table 4.

Table 4. Equipment used in the physicochemical analysis of fuel samples

No	Device/ Equipment	Measurement range	Accuracy
1	Bomb calorimeter BCY-1A	-	≤0.2% (Grade C), (RepeatabilityError)
2	Viscometer, Model VS-R	-	±0.1%
3	Density meter, DMA 4100M	0 to 3 g/cm^3	0.00005 g/cm3(Repeatability Error)
4	FT-IR Analyzer, Brüker Alpha	-	-
5	Flash Point by Pensky Martens Closed Cup Tester k16270	>25°C	-

III. RESULTS AND DISCUSSION

A. Biodiesel Production

Croton oil has an acid value of 7 g KOH/mg. Various samples were prepared to determine the best ratio of reactants that gives an acceptable yield that is economically viable. Table 5 shows the various samples, their oil to methanol ratio, and corresponding yield. Biodiesel yield was evaluated using Eq. (1).

$$\text{Yield} = \frac{\text{mass of biodiesel produced}}{\text{mass of the raw oil}}$$

Table 5. Samples and corresponding biodiesel yield

	SAMPLE 1	SAMPLE 2	SAMPLE 3
Croton oil (ml)	100	100	100
Methanol (ml)	100	200	300
Oil to methanol volume ratio	1 :1	1 :2	1 :3
Sodium hydroxide (NaOH) (g)	1	1	1
Mass of crude biodiesel*	85.5	120	239
Mass of clean biodiesel	84.5	87	87.5
YIELD	0.845	0.87	0.875

Based on economic viability, sample 1, which has a Croton oil to methanol ratio of 1:1, was selected as the most suitable oil to methanol ratio since a lower quantity of methanol is required while the yield is comparable to that of other ratios with a higher quantity of methanol. The yield recorded is also comparable to the yield (88%) reported by Kafuku and Mbarawa [6].

B. Chemical Composition Analysis

Raw Croton oil, Croton methyl ester (B100), and diesel were all subjected to Fourier Transform Infrared (FT-IR) analysis. The FT-IR spectrum for the Croton oil and methyl ester is compared against that of diesel to show the difference in the dominant groups for the fuels, as shown in Fig. 5.

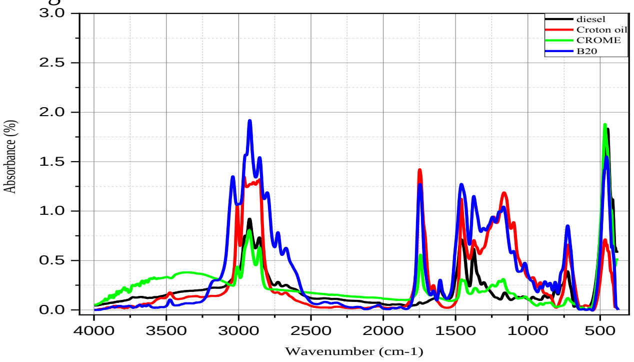

Fig. 5 FTIR spectrum of diesel, raw Croton oil, CROME, and B20

There is no large absorption band between 3800 and 3000 cm^{-1} in the single bond area (2500-4000 cm^{-1}) in the FT-IR spectra for raw Croton oil, indicating the absence of hydrogen bonds [15]. Other peaks were observed between 3150 and 3000 cm^{-1}, implying the existence of unsaturation (C=C-H) and/or aromatic ring[16]. Narrow bonds below 3000 cm^{-1}, with the predominant absorptions around 2935 and 2860 cm^{-1}, as well as absorptions around 1470 and 720 cm^{-1}, indicated the presence of long linear aliphatic chains[17]. Peaks between 2700 and 2800 cm^{-1} indicate the existence of aldehyde, whereas the absence of a peak in the range (2000-2500 cm^{-1}) shows that the substance lacks a C≡C triple bond. [17].

Peaks in the range of 1500-2000 cm^{-1}, especially around 1700 cm^{-1}, indicate the presence of carbonyl double bonds, which can come from ketones, aldehydes, esters, or carboxyl compounds. [18][19]. The lack of a peak around 1600 cm^{-1} suggests that there is no C=C bond in the oil [20]. A strong signal was found at about 1500 cm^{-1} in the fingerprint region (1500-600 cm^{-1}), indicating the presence of aromatic rings and thiols and thio-substituted compounds below 1000 cm^{-1} [16].

Based on the above interpretations, several conclusions can be drawn. One possible conclusion is that the raw Croton oil has no hydrate component. It has aldehydes-related components, with no double or triple bond in the material. The transesterification process led to the formation of new compounds, which reduced the concentration of amine salt, alkyl, alkene, alkane, and other compounds, causing a reduction in the peaks at 2800 cm^{-1} and an increase in the magnitude of the peak at 500 cm^{-1}. The residual methanol in the produced biodiesel is responsible for the broad absorption band in the range of 3200-3600 cm-1 in the case of B100 [21]

The FTIR spectrum for diesel fuel and CROME seem to match in their compositions. The blend in the proportion of 20% CROME and 80% diesel (B20) shows an increase in all peaks along the spectrum.

Fig. 6 and 7 show the FTIR spectra for biodiesel blends with nanoparticles added in varying proportions. The addition of graphene and graphene oxide in different dosing levels (25, 50, and 100 ppm) is seen to affect the composition of the mixture (B20), as depicted by the change in magnitudes of some of the peaks. By increasing the amount of graphene, the peaks along the spectrum are generally reduced. Beyond 50 ppm, the peaks remain relatively constant. However, the addition of graphene oxide is observed to significantly reduce the peaks all along the spectrum. This might be due to the composition of the nanoparticles, but the further investigation should be considered.

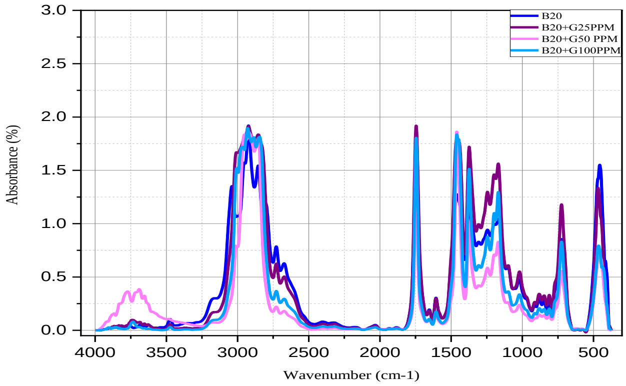

Fig. 6 FTIR spectrum of B20, B20+G25, B20+G50 and B20+G100ppm

* Crude biodiesel: biodiesel from the separation process before washing and drying

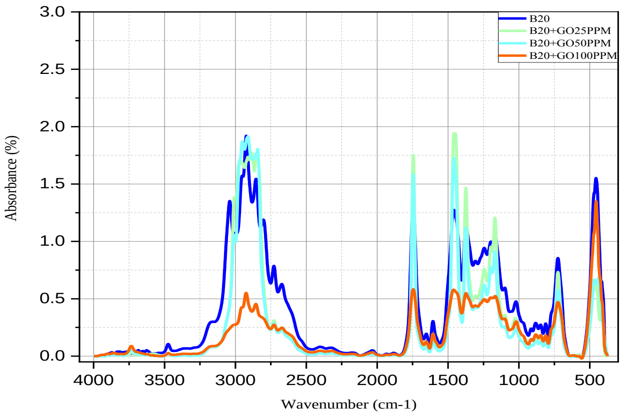

Fig. 7 FTIR spectrum of B20, B20+GO25, B20+GO50 and B20+GO100ppm

C. *Physico-chemical Properties Test*

The physicochemical properties, including density, viscosity, calorific value, and flashpoint of the CROME (B100) were determined, and are presented in Table 6. The properties are compared to those of diesel fuel as well as the EN14214 and ASTM D6751 biodiesel standards. Furthermore, the physicochemical properties of B20, B20+G25 ppm, B20+G50 ppm, B20+G100 ppm, as well as B20+GO25 ppm, B20+GO50 ppm, B20+G100 ppm are investigated to see how graphene and graphene oxide nanoparticles affect the physicochemical properties of the fuel mixes.

a) Density and Viscosity

The density and viscosity of the various fuel samples are displayed in Fig. 8 and 9. The density and viscosity of neat biodiesel (B100) are found to be significantly higher than those of diesel. However, mixing biodiesel with diesel up to 20% biodiesel in the fuel mixture (B20) results in a significant reduction in the density and viscosity of the biodiesel, bringing it closer to that of diesel. The addition of graphene nanoparticles had a minor impact on the fuel blend's density and viscosity.

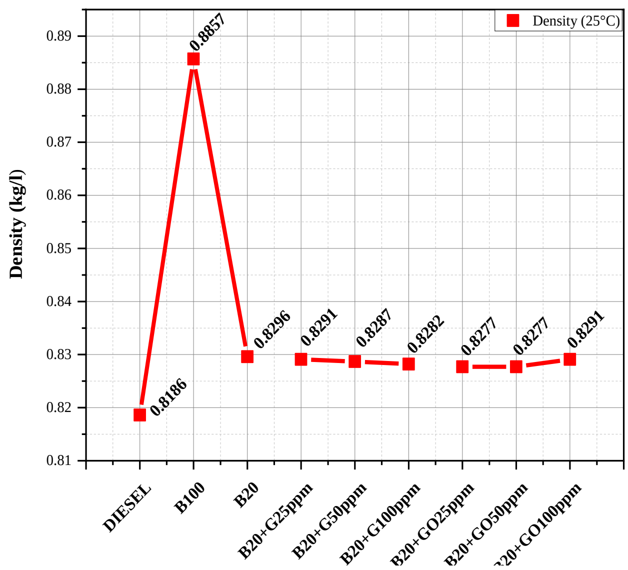

Fig. 8 Density graph

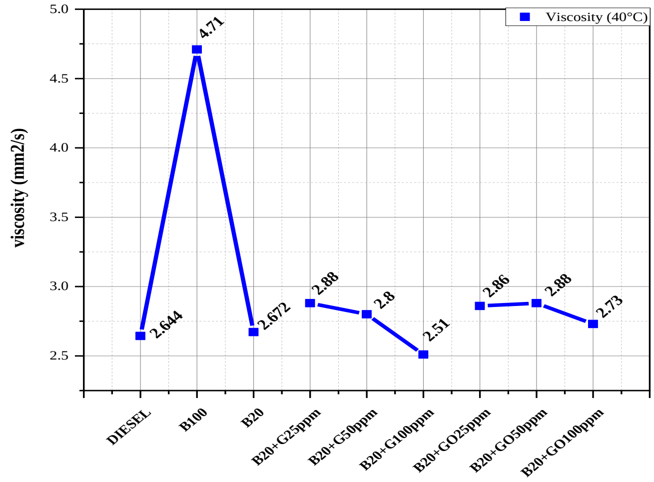

Fig. 9 Viscosity graph

b) Calorific Value and flash point

Fig. 10 and Fig. 11 show the calorific value and the flash point of the samples, respectively. The calorific value of B20 improved compared to that of B100. By adding the nanoparticles, the calorific value improved from 39.41 MJ/kg to 40.63 MJ/kg and this is due to the energy content in the nanoparticles. Flash point is defined as the lowest temperature where a combustible material can ignite by an external ignition source and it is an important parameter for safety during storage, handling and transport [20]. The flash point of the biodiesel is slightly below ASTM D6751 standard but fits EN 14214 standard and high compared to diesel fuel. This indicate that diesel fuel is more volatile and flammable compared to the biodiesel due to the difference in the chemical composition of both. It is also evident that addition of nanoparticles to the fuels improve the flashpoint significantly in some cases, indicating that nanoparticles could considerably influence combustion characteristics of the engine. Graphene and graphene oxide have the opposite effect on the blend. While B20+G25ppm increased the flash point, B20+GO25ppm reduced it. The reason for the observed results is not clear and further investigation is necessary.

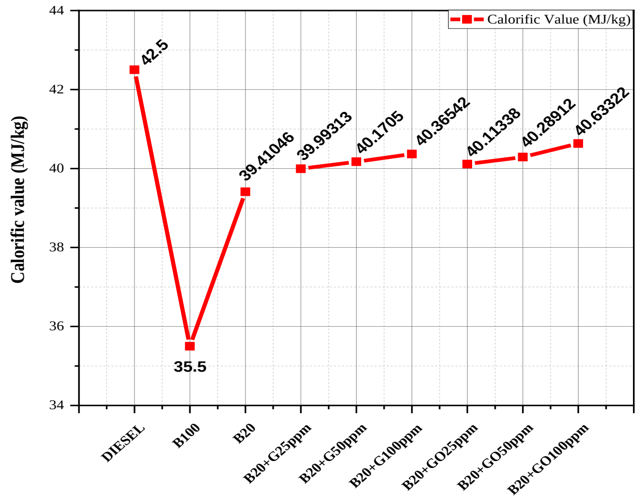

Fig. 10 Calorific value graph

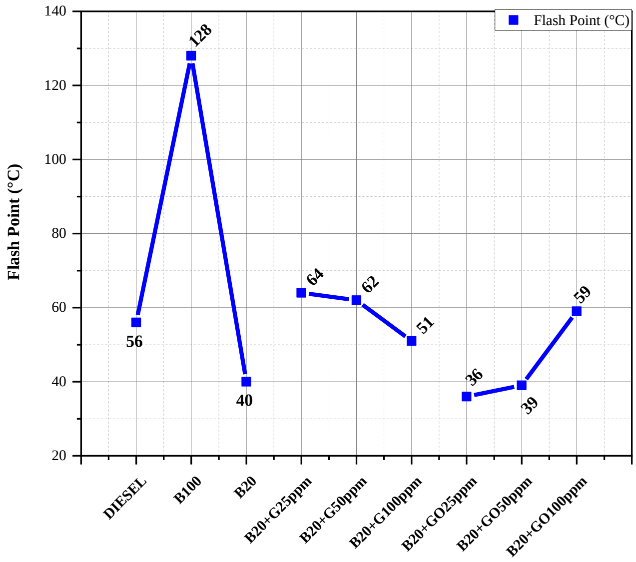

Fig. 11 Flashpoint graph

The physicochemical property data of all the samples are presented in Tables 6 and 7, as obtained from the test.

Table 6. Fuel properties of diesel and biodiesel fuels

	Diesel (D100)		CROME	EN14214/ ASTM D6751		
	Value	Method	(B100)	Min	Max	Method
Density at 25°C(g/cm^3)	0.8186	IS1448	0.8857	0.860/ -	0.900 / -	ISO3675 / -
Viscosity at 40°C (mm² s^{-1})	2.644	ASTM D445	4.71	3.5 / 1.9	5 / 6	ISO3104 / D445
Calorific value(MJ/kg)	42.5	IS:1359-959	35.5	- / -	- / -	- / -
Flash point (°C)	56	ASTM D2500	128	>101/ 130	- / -	ISO CD3679e / D93

Table 7. Fuel properties of samples with Graphene and Graphene oxide nanoparticles

	B20	B20+G25ppm	B20+G50ppm	B20+G100ppm	B20+GO25ppm	B20+GO50ppm	B20+GO100ppm
Density at 25°C (g/cm^3)	0.8186	0.8857	0.8296	0.8291	0.8287	0.8282	0.8277
Viscosity at 40°C (mm² s^{-1})	2.672	2.88	2.80	2.51	2.86	2.88	2.73
Calorific value (MJ/kg)	39.41046	39.99313	39.41046	39.99313	40.1705	40.36542	40.11338
Flash point (°C)	40	64	62	51	36	39	59

IV. CONCLUSION

Biodiesel was made from Croton oil using the transesterification process, and its physicochemical parameters, such as density, viscosity, and calorific value, were compared to those of fossil diesel. Properties of diesel-biodiesel blends and blends with nanoparticles (Graphene and Graphene Oxide) were also investigated to establish the effect of nanoparticles on biodiesel properties. From the biodiesel production results, it is observed that the factors that affect biodiesel production are oil to methanol ratio, reaction temperature, reaction time, the stirring frequency, and the amount of catalyst. Best production conditions were recorded as Croton oil to methanol ratio of 1:1, stirring frequency of 310 Hz, temperature of 60-65°C, and reaction time of 1 hour. The physicochemical properties of the Croton methyl ester, such as density, viscosity, calorific value, are found to lie within the range for biodiesel standard ASTM D6751 and EN 14214. The flashpoint is slightly below the ASTM D6751 standard but fits the EN 14214 standard. Additionally, the FTIR test revealed loose relation in the chemical composition of CROME and diesel.

The overall properties of B20 are comparable to diesel fuel, indicating that the B20 blend can be utilized as fuel for diesel engines. The addition of graphene and graphene oxide in different dosing levels (25, 50, and 100 ppm) has a marginal effect on the physicochemical properties of the blend (B20) but slightly increases the calorific value of the fuel.

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