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Quality assessment of Biodiesel produced from *Hibiscus mutabilis* Seed

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Abstract: *This research studied the potentials of the seed oil of Hibiscus mutabilis for biodiesel production. The oil was extracted using a soxhlet extractor and n-hexane as a solvent before being transesterified into biodiesel. The results obtained showed a low yield of 17.6 ± 0.1 % for the extracted oil. The oil showed a high acid value of 14.02±0.09 mgKOH/g, which indicated high free fatty acid content and the percentage yield of the biodiesel produced was 77.38 ± 0.5%. Fuel properties (color, cloud point, pour point, sulfur content, kinematic viscosity, and specific gravity) determined showed compliance with American Standard Testing and Material (ASTM) and European standard specifications. The profile of methyl esters showed that unsaturated linoleic acid methylester was dominant. The results suggested that* Hibiscus mutabilis *seed oil possesses some properties that were suitable for biodiesel production.*

Keywords: *Biodiesel, methyl esters, Fatty acid composition, transesterification, Hibiscus mutabilis*

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1.0 Introduction

Oil exploration is causing global warming and environmental deterioration, prompting the development of alternative energy sources to petroleum-based fuels. Biofuel, such as biodiesel, are gradually receiving significant attention in the energy sector because of their potential as substitutes for fossil fuels. Biodiesel is a sulfur-free, non-toxic, clean-burning, ester-based oxygenated fuel that is renewable and biodegradable (Mosarof *et al.*, 2016). It can drastically reduce environmental pollution and global warming that probably characterizes some of the fossil fuels (Bhuiya *et al.*, 2016; Ramos *et al.*, 2017). The basic steps in biofuel production include pretreatment, alcohol catalyzed mixing, esterification and transesterification, separation of the product and purification of the biodiesel (Anwar *et al.*, 2018; Garg *et al.*, 2021; Jain and Sharma, 2014; Mitelbash and Remscimdt, 2005).

Because of their prospect, several edible and nonedible plant oils have been investigated and applied for the production of biodiesel. For example, Jatropha plant seed (Banković-Ilić *et al*., 2012), palm oil, olive oil, rapeseed oil, and castor oil (Brännström *et al*., 2018) and others. Hasni *et al*. (2018) has described the production of biodiesel using Hibiscus *sabdariffa* seeds oil as a feedstock and the yield obtained was relatively reasonable. The catalyst used for the synthesis was CaO nanoparticles from the eggshell. The study initiated hope on the use of this plant seed as feedstock for future biodiesel production. Several factors may create differences in yield such as the oil content and other parameters. In continuation of the investigation need of this plant as a source of biodiesel, the present work seeks to produce and analyze biodiesel using *Hibiscus mutabilis* seed oil (as feedstock) and KOH (as a catalyst).

2.0 Materials and method

2.1 Sample collection and preparation

Samples used for the study were collected from dried seeds of *Hibiscus mutabilis* in Samaru market in Zaria within Kaduna metropolis, Nigeria. The research was carried out in Ahmadu Bello University, Zaria Sabon Gari Local Government Area of Kaduna State, Nigeria. The seeds were peeled to obtain the kernels, which were air-dried and pulverized to a fine powdered form and stored in an airtight plastic container.

2.2 Extraction procedure

About two hundred grams (200 g) of air-dried and pulverized plant seeds of *Hibiscus mutabilis* was weighed out and packed into a thimble, which was in turn placed into a Soxhlet extractor.

Extracting solvent (normal hexane (500 cm^3) and anti-bumping chips were placed into 1000 cm^3 round bottom flask and heated on a heating mantle at 70 ◦C. The extraction was allowed to continue until the solvent was clear. The solvent in the round-bottomed flask was collected and dried using a rotary evaporator at 40 °C. The process was repeated to obtain the mean percentage extraction and enough oil for further analysis (Takadas and Doker, 2009). The oil content was evaluated using the following equation

$$\text{Oil content} = \left(\frac{Weight\ of\ the\ oil}{Weight\ of\ sample}\right) \times 100 \quad (1)$$

2.3 Physicochemical analysis of oil

various physicochemical analyses were conducted to evaluate the quality of each of the oil samples. The oil physicochemical properties were determined following standardized American Society of Testing and Materials (ASTM) test procedures. The physicochemical parameters to be analyzed were specific gravity, Iodine value, saponification value, acid value, peroxide value, color, and free fatty acid.

The specific gravity of the oil was evaluated by using a density bottle. The bottle was filled with water and the difference in weight of the bottle before and after illing with water was recorded as the weight of the water. The weight of an equal volume of oil was also recorded and the specific density was calculated using equation 2.

$$SG = \frac{\textbf{Weight of oil}}{\textbf{Weight of equal Water}} \quad (2)$$

The saponification value (SV), the acid value (AV), the peroxide value (PV) and the iodine value (IV) were evaluated using the AOAC (2003) methods

50 ml of alcoholic KOH was added from a burette to a volumetric flask containing 5 cm^3 of each oil, The content was titrated to the phenolphthalein endpoint using 0.5 M of HCl as the titrant. A blank system was also titrated and the saponification value was evaluated using equation 3. (AOAC, 2003)

$$SV = \left(\frac{56 \times N(V_0 - V_1)}{W}\right) \quad (3)$$

where; V_0 is the volume of the solution used for the blank test, V_2 is the volume of the titrant, N is the normality of the HCl and W is the mass of the sample.

In the estimation of the acid value of the oil, 2 cm^3 of the sample was weighed into a 250 cm^3 conical flask and made to dissolve in 25 cm^3 of alcohol. The content was titrated with alcoholic KOH to a phenolphthalein endpoint. Blank titration was performed and the acid value was estimated from the titre value (Ekop *et al*., 2007).

2.4 *Biodiesel production*

2.4.1 Esterification

50 g of *Hibiscus mutabilis* oil was measured into a conical flask using a weighing balance. The conical flask containing the oil sample was placed on a magnetic stirrer and agitated to stabilize the system. The system was heated to a temperature of 60 °C 143.8 g of methanol and 3.2 g of sulphuric acid were added as the heating continue.

2.4.2 Transesterification

After the esterification reaction, further esterification was also carried out. Component KOH 40 cm^3 of *Hibiscus mutabilis* extract was poured into a round-bottomed flask that was immersed in a water bath to attain a set temperature of 55 °C to attain After equilibration, 4 cm^3 of anhydrous methanol and 0.4 g potassium hydroxide (KOH) was added to the flask and the condenser was fitted. The solution was placed on a magnetic stirrer and agitated at 60 °C for for1 hour. The mixture was finally allowed to stand in a separating funnel for 1 hour and the lower layer (glycerol) was tapped off while the upper layer gave the biodiesel.. (Shika and Chauhan, 2012).

2.5 Analysis of biodiesel

The density of the biodiesel was measured using the ASTM (2010) method. The kinematic velocity of the biodiesel was evaluated using the ASTM (2010) method. The ASTM D6892-03 and ASTM D-1500 methods were used for the determination of pour point and colour respectively

2.6 FTIR and GCMS analysis

The functional group of the biodiesel oil was analyzed using an FTIR machine (Agilent Technology Fourier transform infrared spectroscopy (FTIR) machine).

The fatty acid composition of *Hibiscus mutabilis* product was evaluated using Shimadzu brand QP2010 model (Kyoto, Japan) Gas Chromatograph (GC) system equipped with DB-5MS capillary column (30 m × 0.32 mm × 0.25 µm) and compared with the most common feedstock used in the production of biodiesel. After sample injection, the temperature was augmented up to 70 °C for 1 min at the beginning And progressively increased to 120 °C at heating rates of 20 °C/min (held for 2 min), 180 °C at 10 °C/min (held for 3 min) and 240 °C at 5 °C/min (held for10 min). Other adjusted operation conditions were injector temperature of 250 °C, split ratio (1:10), carrier gas (helium), gas flow rate (1.5 mL/min), ionization mode used at electronic impact (70 eV), and the volume of injected sample (1 µL) (Yesilyurt and Cesur, 2020)

3.0 Results and Discussion

The physicochemical properties of the *Hibiscus mutabilis* seed oil (Table 1) show a percentage oil content of 17.6 ± 0.52%. The oil content in is lower than the oil contents of *Irvinia gabonesis* (81.94 %) reported by Adekunle *et al.,* (2016) and that of *C. pepo* (52.8%) reported by Bwade *et al*., (2013). The difference is due to differences in species since different plant seeds have different oil content (Eddy and Ekop, 2006; Eddy *et al.,* 2011; Gandhi *et al*., 2021) According to the Food and Agricultural Organization (FAO), seeds with lipid content of more than 17% are oil seeds that are most used as a feedstock for biodiesel synthesis, as described by Akinoso and Raji (Akinoso and Raji, 2010). In general, a poor oil yield indicates that the oilseed may be scarce for oil production.

The specific gravity of *Hibiscus mutabilis,* yields 0.89±0.00. This suggests that the oils are less dense than water and that they do not contain any heavy components. The results of this study were found to agree with those reported for 0.91, for *Arachis hypogeal*, as reported by Motojesi *et al.*, (2011) but lower than the values reported for *Anarcardium occidentalis* (0.96) by (Ren, 2010). Specific gravity is a critical quantity to assess since it determines the energy density (specific energy) of a gasoline fuel (Atabani *et al.*, 2012),

Table. 1: Physicochemical properties of the seed oil (Mean ± SD)

Properties	***Hibiscus mutabilis***
Specific gravity	0.89±0.00
Acid value (mg KOH/g)	14.02±0.09
Iodine value	23.69±0.34
S.V (mg KOH/g)	126.23±0.73
Viscosity (mm/s)	5.77±0.09
Peroxide Value (Meq /Kg)	5.51 ± 0.61

The acid value of *Hibiscus mutabilis* was 14.02±0.09 mgKOH/g. The lower the acid value, the lower the free fatty acid, the more suitable, the oil for the transesterification process. The acid value of the oil was higher in comparison to the acid value of rubber seed oil of 8.17 mgKOH/g reported by Eka *et al* (2010), which indicated that over time, the oil will become unstable, and it will not be safeguarded against rancidity or peroxidation (Mabaleha *et al.*, 2007).

Hibiscus mutabilis had a saponification value of 126.23±0.73 mgKOH/g, which was lower than the cocos *nucifera* oil (246 mgKOH/g) studied by (Eka *et al.*, 2010). Biodiesel generated from oil with a high saponification value, on the other hand, produces exhaust pollutants when burned in an engine (Bwade *et al.*, 2013).

The iodine value of *Hibiscus mutabilis* was 23.69±0.34 gI2/100g, which was lower than the iodine value of castor oil (84.8 gI2/100g) as reported by Aremu *et al.*, (2015). This suggests that the oil may be suitable for biodiesel production since low iodine value of vegetable oil produces biodiesel with high cloud and pour points; higher cloud and pour points mean poor engine performance in cold temperatures (Eka *et al.*, 2010).

In order to achieve the gasoline standard, viscosity is an important property that must be regulated in oil. The viscosity for *Hibiscus mutabilis* was 5.77±0.09 mm^2/sec, which was lower compared to the viscosity of *Cucurbita pepo* oil (93.65 mm^2/sec) according to Bwade *et al* (2008). Low viscosity oil produced a sloppy effect (Yusup and Khan, 2010).

The peroxide value of *Hibiscus mutabilis* was 5.51 ± 0.61 Meq/kg was lower than the value of 6.61 meq/kg oil reported by Yusuf *et al.*, (2015) for *Albizia julibrissin*. This showed that the oil will have a high level of deterioration when attacked or exposed to oxygen since peroxide value indicated the level at which deterioration will take place as a result of oxidation owing to the availability of oxygen during storage (Atadashi *et al.*, 2012).

3.2 Fuel properties

The fuel property of the biodiesel produced from *Hibiscus mutabilis* is shown in Table 2. The percentage biodiesel yield was recorded to be 77.38± 0.06 %., which was lower than the the percentage yield reported for the oilseeds oil from *Lagenaria vulgaris* seeds with 96.52% (Nick and Greg, 2008).

Table 2: Fuel properties of *Hibiscus mutabilis* Methyl ester (Mean ± SD)

Properties	***Hibiscus mutabilis***
Biodiesel yield (%)	77.38
Specific gravity	0.86±0.00
Pour point (^{0}c)	-4.6±0.08
Cloud Point (^{0}c)	6.83±0.09
Kinematic viscosity (@40^{0}C)	3.33±0.12

The specific gravity of the Hibiscus mutabilis methyl ester was 0.86±0.00 g/cm3 this is lower than the specific gravity of biodiesel produced from *Lagenaria vulgaris* with a specific gravity of 0.8879g/cm^3 as reported by Sokoto *et al.*, (2013). This showed that the biodiesel produced could be securely handled and stored.

In the management and storage of fuels, it was a key physical property (Nurhazirah, 2013).

The pour point of liquid fuel is the lowest temperature at which it loses its flow qualities. For the cold flow process, the pour point is a crucial characteristic. The pour point of the *Hibiscus mutabilis* methyl was -4.6±0.08 ^oC.

The cloud point of the *Hibiscus mutabilis* was 6.83±0.09 ^oC, cloud point test characterized the low-temperature operability of diesel fuel. In general, a fuel with a high cloud point has limited utility in cold climes. Nonetheless, the results were within range of ASTM standards, though they are very high. In general, a fuel with a high cloud point has limited utility in cold climes. Nonetheless, the results were within range of ASTM standards, though they are very high.

The most important methyl ester characteristic is Kinematic viscosity, which affects fuel injection and sprays atomization, particularly at low temperatures [19]. The kinematic viscosity of the *Hibiscus mutabilis* methyl esters is 3.33±0.12 which corresponds to biodiesel specification for both ASTM and European Standard limits, which indicated the presence of short-chain unsaturated methyl fatty acid esters and was likely to produce less deposit when burnt in combustion engines (Sokoto *et al.*, 2013).

Fig. 1 shows the IR spectra of *Hibiscus mutabilis* methyl esters, which are mono-alkyl ester of fatty acids. The existence of an intense band due to C=O stretching of the methyl ester and O-CH_3 group in the spectra is confirmed. The transesterification process was validated by the absorption band ascribed to the OCH_3 group (Sokoto *et al.*, 2013). The biodiesel's FTIR profile shows some prominent absorption bands at 1738 cm-^{1}, 1176 cm^{-1}, 2938 cm^{-1}, and 1446 cm^{-1}, which are consistent with results obtained by Sanford *et al*. (2009).

The GCMS spectrum of the produced biodiesel shown in Fig. 2 reveals the presence of s pome notably, at retention time, 15.660, 16.349, 17.441, 18.064 and 18.272 minutes. Identified compounds and their composition are shown in Table 3. The major constituents of the biodiesel are unsaturated fatty acid (71.29%) compared to saturated components (28.71%). The distribution ratio are as shown in Table 3. Redel-Macías *et al*. (2012) have produced biodiesel with a high percentage of saturated components and stated that their product burnt effectively and was characterized by octane number. Therefore, the composition of the produced biodiesel is favourable.

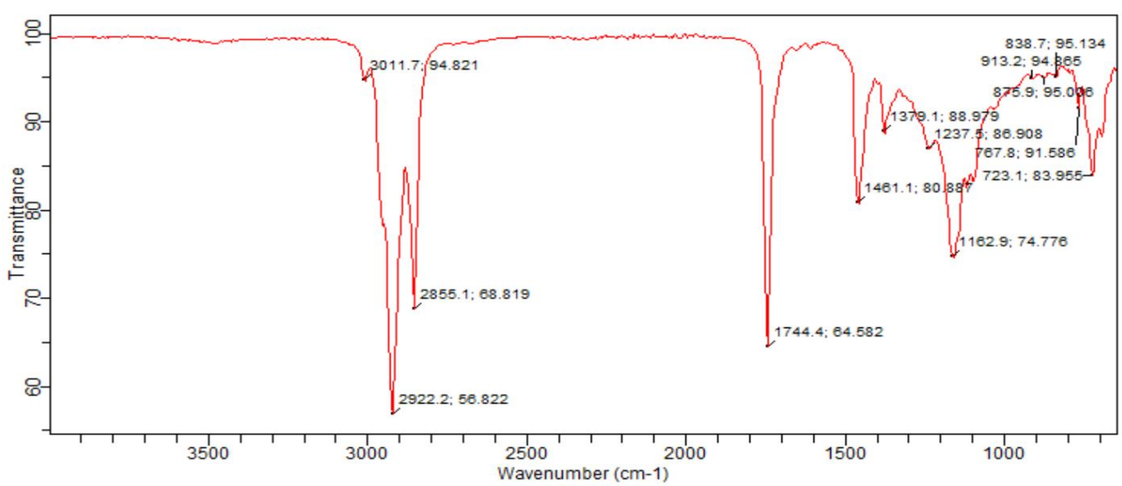

Fig 1: FTIR Spectra for *Hibiscus mutabilis*

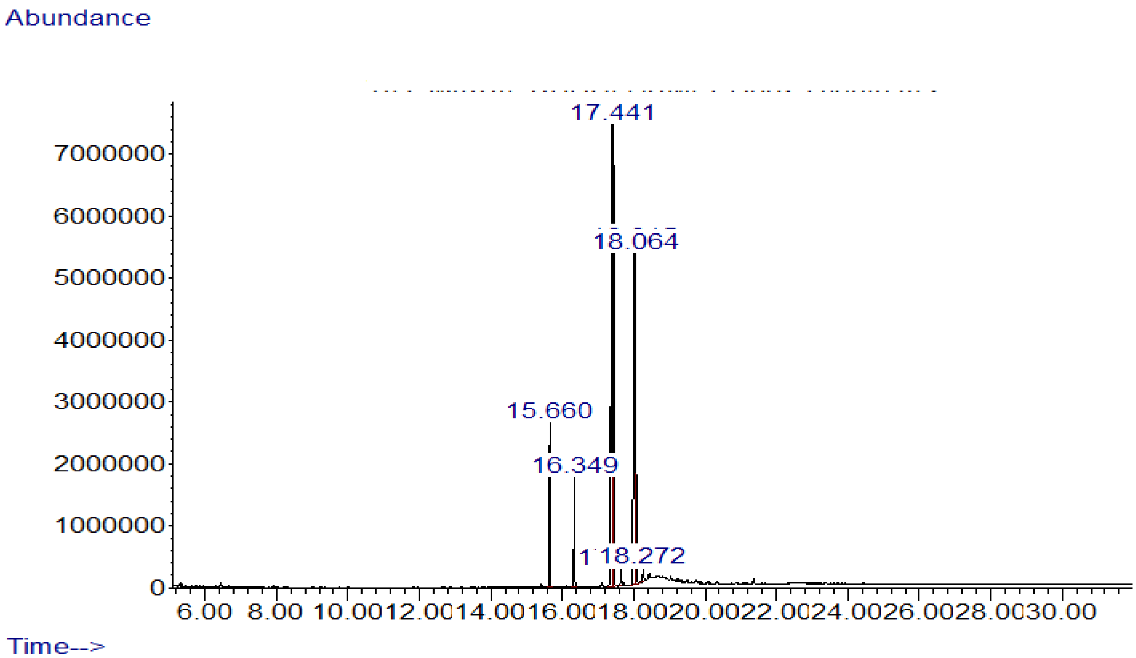

Fig 2: GC – MS spectra of *Hibiscus mutabilis* Biodiesel

Table 3: GCMS data for the produced biodiesel

Fartty acid	Molecular formular	C (%)
Hexadecanoic acid, methyl ester	$C_{16}H_{32}O_2$	7.68
9, 11 Octadecadienoate acids	$C_{18}H_{31}O_2$	9.33
Linoleic acid	$C_{18}H_{32}O_2$	10.34
Ethyl oleate	$C_{20}H_{38}O_2$	14.00
9, 12 Octadecenoic acid, methyl ester	$C_{18}H_{24}O_2$	29.90
Octadecanoic acid, methyl ester	$C_{18}H_{36}O_2$	0.93
Dodecanoic acid, methyl ester	$C_{12}H_{24}O_2$	5.32
Heptadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	0.78
9, Octadecenoic acid, methyl ester	$C_{18}H_{34}O_2$	21.72
Saturated		28.71
Unsaturated		71.29

4.0 Conclusion

This study investigated the esterification and transesterification of Hibiscus mutabilis seeds using potassium hydroxide to produce biodiesel. Overall, 77.8 percent of biodiesel was produced. Biodiesel's properties were determined using a traditional approach. All test parameters evaluated met or exceeded ASTM requirements. Biodiesel's properties were determined using a traditional approach. All test parameters evaluated met or exceeded ASTM requirements. As a result, Hibiscus mutabilis is an economically viable, environmentally acceptable, and non-edible feedstock for the generation of high-quality biodiesel.

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There is no bridge of ethics and consent to participate in this manuscript based on the existing laws.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data public

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There is no competing interests

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