

Characterization of *Cassia Tora* Seed (CTS) Oil-Based Biodiesel-Diesel Blends

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Abstract. The paper reported on the characterization of CTS oil-based biodiesel-diesel blends. The study's primary objective are the physicochemical characterization of oil and biodiesel from *Cassia Tora* seed (CTS) oil, characterization of CTS oil-based biodiesel using FTIR and GCMS analysis. Extraction revealed the presence of 8.8% oil in *Cassia Tora* seed. The CTS biodiesel's viscosity was found to be 0.9092, which is well above the petroleum diesel.

The physicochemical characterization showed that 5, 12.5 and 20 % CTS oil-based biodiesel-diesel blends are within acceptable limit for combustion engine without modification having viscosities of 0.8744, 0.8764, and 0.8787 respectively while 100% CTS oil-based biodiesel would require upgrading to meet up with the recommended standard for fuel used as diesel in internal combustion engines.

FTIR and GCMS analysis showed functional groups and characteristic peaks of fatty acids and methyl ester with carbon range from C₁₃-C₁₇, C₁₆ constituted the major Fatty acid group and contained 61.69 % Monounsaturated Fatty Acid (MUFA), and 38.31 % Saturated Fatty Acid (SFA) giving a 94.3% biodiesel yield.

Keywords: characterization; oil; blends; diesel; physicochemical; biodiesel; fatty acid; cassia tora; seed.

INTRODUCTION

The chief chemical constituents of *Cassia Tora* include cinnamaldehyde, gum, tannis, mannitol, coumarins, and essential oils. Phytochemical analyses of *Cassia* plant extracts and callus extracts contain anthrone, flavonoids, glycoside, and anthracene derivatives. The leaves have approximately 72% water, 10% petroleum ether extract, 0.90% ether extract, 1.60% absolute alcohol extract and 20% watery extract. It also contains sugars, resins, and mucilage, among other constituents [20].

The plant as a whole and specific parts such as the roots, leaves and seeds have been widely used in traditional Indian and South Asian medicine [3]. The plant's edible amount varies from 30 to 40 %, and the young leaves can be cooked as a vegetable while the roasted seeds could serve a good substitute for coffee and its flowers are added to food in Sri Lanka [3]. It is used as a natural pesticide in

organic farms, and its powder is most commonly used in the pet food industry, and it is mixed with guar gum for use in mining and other industrial applications. The seeds and leaves are also used for skin disease treatment, and its sources can be utilized as a laxative. In the Republic of Korea, it is believed to rejuvenate human vision. The tea is believed to have created a new term "coffee-tea", because of its weird but vibrant taste and its coffee aroma. The tea is made from 100 % *Cassia Tora*, with no artificial colouring and no caffeine, and could be a healthier substitute for coffee and sodas. It has been used to treat skin diseases such as leprosy, ringworm, itching and psoriasis, and snakebites due to its external germicide and anti-parasitic character [3].

Cassia Tora seed is one of the oil seeds discovered worldwide [18]. The choice of oil-yielding plant species depends mainly on a thorough understanding of cultivation characteristics like soil

character, salt content, nature and content of organic matter, and pH that influence the plant's oil yield. But the overall suitability of extracted crude vegetable oils for technical purposes, the acquaintance of oil content, physical properties, and oils' chemical properties are essential.

With the advent of cheap petroleum, appropriate crude oil fractions were refined to fuel while diesel fuels and diesel engines evolved together. Between 1930–1940 vegetable oils were used as diesel fuels but usually only in emergencies. Due to crude oil prices, limited resources of fossil oil and environmental concerns, there has been a renewed focus on vegetable oils and animal fats to make biodiesel fuels. Continued and increasing use of petroleum will intensify local air pollution and magnify the global warming problems caused by CO₂. In a particular case, such as the emission of pollutants in the closed environments of underground mines, biodiesel fuel has the potential to reduce the level of contaminants and the level of potential or probable carcinogens [17].

Currently the industrial biodiesel production bases almost exclusively on the chemical trans-esterification of triglycerides (TAGs) from vegetable oils employing methanol. However, other short-chain alcohols like ethanol, propanol could be used as well [16]. Among all the feedstock vegetable oils (both edible and non-edible) are a most promising feedstock for the biodiesel production since they are renewable and can be produced in large scale. Still, the use of edible oil as feedstock may cause some problems such as the competition with the edible oil market, which increases both the cost of edible oils and biodiesel. To overcome these disadvantages, most of the research focuses on non-edible oils that are not suitable for human consumption because of some toxic oil components.

MATERIALS AND METHODS

Materials used for this finding are Cassia Tora Seeds, reagents and apparatus/equipment. The seeds were collected at different locations within Bauchi town.

All chemicals and solvents used were analytical grades from Sigma Aldrich, England. These are acetone, chloroform, concentrated sulfuric acid, diethyl ether, ethyl acetate, ethanol, hydrochloric acid, iodine monochloride, n-hexane, petroleum ether, phenolphthalein, potassium hydroxide, sodium hydroxide, sodium thiosulphate.

The apparatus and equipment used in this study are test tubes, measuring cylinders, 500 ml round bottom flasks, reflux condensers, 250 ml beakers, 50 ml pipette, 5 ml micro burette, paper filter (1.0 mm), Soxhlet extractor, separator funnel, 100 ml burette, spatula, dropper, stirring bit, and other related glasswares. Weighing balance (Melrose pack, Illinois), Gallen Kamp magnetic stirrer, gladden viscometer, Corning pH meter, Bellingham refract meter, Copenhagen digital temperature measurement, Smith stopwatch, TD114 instrumentation unit, TD115 hydraulic dynamometer, GC-MS Shimadzu QP500, RE-52A rotary evaporator, and Madison WI thermo Nicolet analytical instrument.

Methods

1) Cassia Tora Seed Oil extraction (adopted by [22]). Cassia Tora Seeds were ground to obtain 0.037–2.0 mm for proper extraction. 80 g of the ground sample was packed in a paper filter and inserted into the soxhlet extractor used is petroleum ether was used as a solvent. 500 cm³ of the solvent was used on 80 g of the sample for six hours of extraction. At the end of successful extraction, the solvent was recovered using a rotary evaporator. The residual oil was oven-dried at 105 °C for one hour before the sample was weighed for further experimentation.

2) Esterification and trans-esterification. Optimum values for Cassia tora free fatty acid reduction (Esterification) and Cassia tora biodiesel production (trans-esterification) was adopted by [22]. In esterification, the high FFA of CTO was reduced by its pretreatment with methanol/oil ratio (6:1), H₂SO₄ catalyst (1.6 v/v) in 93 min at 45 °C while the trans-esterification Cassia Tora biodiesel was produced using methanol/oil molar ratio (9:1), NaOH catalyst (0.8 w/w) in 120 min at 50 °C.

3) Gas Chromatography-Mass Spectrometry (GC-MS). For GC-MS analysis, 100 L of oil biodiesel was dissolved in 2 mL of dichloromethane (DCM). The sample was analyzed on a Shimadzu GC-MS system model QP500 with a medium polarity capillary column (BPX-5 column (29.4 m by 0.25 mm). The film thickness of 0.25 m) with helium as the carrier gas. One microliter of the sample was injected using splitless injection with injector temperature 300 °C according to a scheme of 50 °C for 2 min with 10 °C/min up to a maximum temperature of 300 °C. The final temperature was held for

10 min. The total runtime for each sample was 37 min. For MS detection, electron ionization with 70 eV was applied, and mass fragments were detected between 40 and 500 m/z. The ion source temperature and transfer line temperature were 200 °C and 300 °C, respectively, after the detector was activated for 5 min.

4) Fourier Transform Infrared (FTIR). All spectra were obtained using an ATR-FTIR of Thermo Scientific (Thermo Nicolet Analytical Instruments, Madison, WI). The spectra were collected at a resolution of 4 cm⁻¹ in the range of 4000–650 cm⁻¹. Each spectrum was rationed against a fresh background spectrum recorded from the bare ATR crystal. Before collecting each background spectrum, the ATR crystal was cleaned with absolute ethanol to remove any residual. Each sample was scanned in triplicate.

5) Biodiesel-diesel blends formulation. To establish blends for diesel engine performance investigation, biodiesel-petroleum diesel fuels were blended by mixing the biodiesel and diesel in different proportions using an in-tank blending method. The most common biodiesel-diesel blends were in the range of 2–30 % biodiesel (i.e. B2-B30) [6]. Many diesel engine original equipment manufacturers (OEMs) approve blends in the range of 2–30 % on the engine without modifications [1]. On this basis, the blend ratios in this report were set within the feasible range (2–30 %) of biodiesel blend for the combustion and engine performance analysis.

6) Physicochemical characterization of Cassia Tora biodiesel-diesel blends. The various blended biodiesel-petroleum diesel was characterized for the combustion and performance characteristics such as kinematic viscosity, specific gravity, flash point, pour point, heating value, fuel consumption analysis, and power rating analysis determining their suitability for use in the diesel engine.

Knowing the saponification and iodine value of the various biodiesel-diesel blends produced, using the correlation given by [21], each blend's cetane number was calculated using equations (1)–(3).

$$\begin{aligned} \text{Cetane Number (CN)} &= \\ &= 46.3 + \left(\frac{5458}{SV}\right) - (0.225 \times IV) \end{aligned} \quad (1)$$

$$\begin{aligned} \text{Saponification Value (SV)} &= \\ &= \left(\frac{56.1 \times B - A \times N}{\text{weight of oil sample used}}\right) \end{aligned} \quad (2)$$

$$\text{Iodine Value (IV)} = \left(\frac{(B-A) \times 100 \times 126.9 \times 2}{(100 \times W)}\right) \quad (3)$$

where B – the volume of standard ethanol potassium hydroxide used in blank titration;

A – volume of standard ethanol potassium hydroxide used in biodiesel titration;

N – normality of standard acid used in the titration;

W – the weight of the oil sample.

Flashpoint analyzer was used for the test. The biodiesel blend was poured into the analyzer copper container to the prescribed mark, and the cover fitted to its position. Heating was carried out using Bunsen burner at the same time stirring of the sample follows, and the temperature at which a clean flash occurred was recorded as the flashpoint.

Kinematic viscosity was carried out using the NDJ-5S Digital Rotary Viscometer and the DBK Mini Magnetic Stirrer/Heater. The biodiesel blend was placed in beakers and the beaker placed on the heater with the viscometer's piston placed inside it. As the piston of the viscometer rotates in the beaker and the biodiesel blend heated. A thermometer placed at the beaker's side to monitor temperature and readings was taken from the viscometer at 40 °C and 100 °C [23]. The same procedure was repeated for all other blend samples.

Pour point test was carried out in the laboratory using a bath, test tubes and a negative thermometer. Measured quantities of B5, B12.5 and B20 biodiesel blend samples were poured into a medium-sized test tube and placed in the ice bath. The temperatures were checked periodically at 30-sec intervals for flow characteristics with the thermometer to check for the temperature. The temperature at which the B5, B12.5 and B20 biodiesel blend samples start to coagulate was determined as its pour point [23]. The same procedure was repeated for all other instances.

10 cm³ of the biodiesel blend was measured in a pre-weighed measuring cylinder. The cylinder and biodiesel blend's weight was measured; the biodiesel blend's importance was obtained by subtracting the cylinder's weight from the weight of the biodiesel blend and cylinder. The specific gravity of the biodiesel blend was obtained using equations (4)–(6) by [23].

$$\rho_{water} = \frac{W_1 - W_0}{V_0}, \quad (4)$$

where W_1 = weight of measuring cylinder + water;

W_0 = weight of empty measuring cylinder;

V_1 = volume of water used.

$$\rho_{blend} = \frac{W_2 - W_0}{V_2}, \quad (5)$$

where W_2 = weight of measuring cylinder + bio-diesel blend;

V_2 = volume of biodiesel blend.

$$\text{Specific gravity} = \frac{\rho_{blend}}{\rho_{water}}. \quad (6)$$

The calorific value is the measurement of heat or energy produced and is measured either as gross calorific value or net calorific value. The calorific values were obtained from a survey of diesel fuel and biofuels.

RESULTS AND DISCUSSION

Oil extracted from CTS was used for biodiesel production with the aid of Soxhlet apparatus and petroleum ether solvent with an oil yield of 8.8 %. Optimized values of the process variables used in the esterification (reduction of FFA in oil) and transesterification reaction are presented in Table 1.

Table 1 – Optimized values of Process variables for esterification and transesterification reaction

Reaction	Methanol Oil Ratio (v/v)	Catalyst (w/w)	Temperature (°C)	Reaction time (min)	Value
Esterification	6	1.6	45	93	FFA: 0.38 mgKOH/g
Transesterifi- cation	9	0.8	50	120	Biodiesel yield: 94.3 %

These optimum process parameters used (methanol-oil ratio of 6:1, acid catalyst of 1.6 v/v and 93 min reaction time) for esterification of the CTS oil and, the methanol-oil ratio of 9:1, the base catalyst of 0.8 v/v and 12 min reaction time for biodiesel production were adopted from [22]. Validation experiments were conducted to validate the reliability of the optimum parameters established by [22] for maximum yield. Table 1 presents the result obtained from the validation study for esterification and transesterification of CTS oil.

Table 1 observed that the FFA of the CTS oil was reduced to 0.38 mg KOH/g from initial value of 2.5 mg KOH/g due to esterification pretreatment of the CTS oil which is comparable to 0.395 mg KOH/g FFA reported by [22] for CTS oil.

This reduction is necessary because high FFA in oil reduced process efficiency of the oil for biodiesel production as it will lead to saponification reaction and decreases yield during biodiesel production, as such oil used for biodiesel product should have FFA < 0.5 mg KOH/g (FFA < 1%) [9, 13]. It was also found that 94.3% biodiesel yield was obtained from transesterification of CTS oil with methanol at the catalyst presence's optimum parameter. The 94.3 % biodiesel yield obtained

from the validation study compared favourably with 95.9 % reported by [22]. The high biodiesel yield from the pretreated oil could be attributed to reducing the FFA content in the oil to FFA < 0.5 mg KOH/g [13, 15]. The validation study showed that the optimum parameter reported by [22] is reliable.

Fourier transforms infrared spectroscopy analysis

The FTIR is a measure of the quantitative and qualitative analysis of the functional group of organic and inorganic samples. The FTIR spectrum for the extracted oil is shown in Figure 1, and the result obtained from the transmittance spectrums is presented in Table 2. From the FTIR impact obtained, as shown in Table 2, the constituent functional groups of the extracted CTS oil are mainly, carboxylic, carbonyl, aliphatic and hydroxyl functional groups of ester and carboxylic acids. The peaks obtain from the FTIR analysis are shown in Figure 1.

Table 3 – GCMS analysis of extracted CTS oil

No	Retention time	Area	Percentage composition	Carbon number	Systematic name	Common name	Molecular Weight
1	11.70	20.63	3.76	C13:0	Tridecanoic acid	Tridecylic acid	228
2	11.86	6.63	1.20				
3	14.14	3.47	0.63	C14:1w5	Tetradecenoic acid	Mysristoleic acid	240
4	15.30	17.71	3.22	C14:0	Tetradecanoic acid	Myristic acid	242
5	15.80	12.63	2.30				
6	17.26	12.69	2.31	Iso C15:0	Iso-Pentadecanoic acid	Iso-Pentadecylic acid	256
7	18.16	105.99	19.32	C16:0	Hexadecanoic acid	Palmitic acid	270
8	18.56	10.93	1.99	C16:1w11			
9	19.28	291.82	53.20	C16:1w7	cis-9-Hexadecenoic acid	Palmitoleic acid	268
10	19.39	32.24	5.87	C16:1w5			
11	19.80	4.71	0.85	Iso C16:0	Iso-Hexadecanoic acid	Iso-Palmitic acid	270
12	20.58	29.55	5.38	C17:0	Heptadecanoic acid	Margaric acid	284

From Table 3, most of the fatty acid constituent is in the range of C16–C17 and constitute 86.61% of the extracted oil's fatty acid content. The MUFA constituent of 61.69% fatty acid and 38.31% SFA in the extracted oil are higher than 43.21% MUFA and 19.37% SFA reported by [5] for CTS oil. Table 11 shows that the main MUFA constituent in the extracted oil is Palmitoleic acid (53.20%) compared to Oleic acid (43.21%) reported by [5] for CTS oil. The difference in the MUFA type in the extracted oil and that reported by [5], could be attributed to factors such as species and strain of the plant, regional and climatic factors, degree of ripeness, harvest and storage method of the seeds, chemical refining process among others.

The validity of the fatty acid that was deduced from the chromatograph was affirmed by comparing it with other reports that have been made on the characterization of the fatty acid composition of the Cassia Tora plant. Five Fatty acids identified from the extracted CTS oil (Table 3) were also reported by [1, 7, 19] for the Cassia Tora plant's leaves and stems.

Physicochemical characterization of diesel, biodiesel-diesel blend and biodiesel

The biodiesel produced from CTS oil and the combinations with petroleum diesel at different ratios were characterized to determine its physicochemical properties for its suitability for combustion engine use. Table 4 presents the physicochemical properties of diesel, biodiesel-diesel blend and biodiesel produced in study.

B0=100% petroleum diesel, B5=5% Biodiesel and 95 % petroleum diesel, B12.5=12.5% Biodiesel and 87.5 % petroleum Diesel, B20=20% Biodiesel and 80% petroleum diesel and B100=100 % Biodiesel.

The specific gravity, a measure of fuel oil's flowability, was determined for diesel, biodiesel-diesel blend and biodiesel. From Table 4, the specific gravity of the petroleum diesel (B0), which is 0.8718 is comparable to those of B5 (0.8744) and B12.5 (0.8764). In contrast, the specific gravity of B20 (0.8787) and B100 (0.9092) is slightly higher than that of petroleum diesel (B0). It can be seen that the specific gravity of B0, B5, B12.5, B20 and B100 are within the recommended EN14214 standard and BIS 15607 international average of 0.86–0.91 particular gravity for biodiesel fuel used in internal combustion engines [12, 15].

Table 4 – Physicochemical properties of diesel, biodiesel-diesel blend and biodiesel

Properties	Unit	Diesel (B0)	B5	B12.5	B20	B100	Standard
S.G	15/4 °C	0.8718	0.8744	0.8764	0.8787	0.9092	0.86–0.91
Colour	ASTM	1.0	2	2.5	2.5	6.5	6 max
Total Sulphur	%wt.	0.137	0.131	0.125	0.118	0.039	0.5 max
Viscosity @ 40 °C	cSt	4.99	4.38	5.13	6.17	20.6	1.9–6.0
Cloud Point	°C	4	-3	-4	-4	7	Varies
BS&W	%Vol.		Trace	Trace	Trace	Trace	-
Flash Point	°C	96	85	80	75		70 min
Cetane Number		44	41	40	40	35	40 min
Calorific Value	MJ/kg	43.2	42.83	42.45	41.7	39.5	38 min

Viscosity, a measure of the fuel spray atomization and fuel system lubrication was evaluated for the diesel, biodiesel-diesel blend and biodiesel samples. Table 4 shows that kinematic viscosity of B0 which is 4.99 cSt is comparable to those of B5 (4.38 cSt), B12.5 (5.13 cSt) and B20 (6.17 cSt) while the viscosity of B100 which is 20.6 cSt was higher. The kinematic viscosity of B0, B5, B12.5 and B20 are within the recommended ASTM 6751 standard of 1.9–6.0 cSt and BIS 15607 international average of 2.5–6.0 cSt while the viscosity of B100 is much higher than the recommended standard for fuel used as diesel in internal combustion engines [12, 14,]. The high viscosity of the B100 indicates the need for blending of the CTS oil-based biodiesel with petroleum diesel to enhance its viscosity.

Flashpoint of a liquid is the minimum temperature at which the liquid gives sufficient vapours to ignite momentarily when a flame of standard dimension is brought near the liquid's surface. It is a measure of safety in the handling of fuel oil. Flashpoint of the diesel, biodiesel-diesel blend and biodiesel samples were also evaluated as shown in Table 4, the flashpoint of B0, B5, B12.5, B20 and B100 are within the recommended ASTM 6751 standard (minimum of 80 °C) and EN14214 standard (minimum of 70 °C) for fuel used as diesel in internal combustion engines [12, 14].

The calorific or heating value measured by the heat released during combustion of a specific amount of fuel was determined for the diesel, biodiesel-diesel blend and biodiesel samples. Table 4 shows that the calorific value, which is a measure of the fuel economy, was defined as 43.2 MJ/kg for B0 (petroleum diesel) while those of the blends are in the range of 39.5–42.83. The calorific value of B0, B5, B12.5, B20 and B100 are high and comparable to recommended ASTM 6751 standard, EN 14214 standard and BIS 15607 international

standard of 38 MJ/kg minimum for fuel used as diesel in internal combustion engines [12, 14]. The calorific value of the B100 (39.5 MJ/kg) reported in this study is comparable to 39.79 MJ/kg [14] and 39.9 MJ/kg [10]. It was observed that the increase in biodiesel blending ratio results in a decrease in the blend's calorific value.

Cetane number, a measure of the ignition, smoking, and emission-quality fuel oil was determined for the diesel, biodiesel-diesel blend and biodiesel samples.

From Table 4, the cetane number of 44 obtained for B0 (petroleum diesel) is higher than those of B5, B12.5, B20 and B100, which are in the range of 35–44.

The obtained cetane number for B0, B5, B12.5 and B20 comparable to the minimum recommended value of 40 by ASTM D-6751 for fuel used as diesel in internal combustion engines while that of B100 is lower [12, 14]. Hence B0, B5, B12.5 and B20 are suitable for use in combustion engines.

Cloud point is a measure of the temperature at which cloudy and wax formation in liquid fuel was observed. The cloud point for diesel, biodiesel-diesel blends and biodiesel samples were determined as shown in Table 4. The cloudy point of 4 °C and 7 °C obtained for B0 (petroleum diesel) and B100. It can also be seen that the cloud point for B5, B12.5 and B20 are much lower than those of B0 and B100. The cloud point of -4 °C obtained in this study for B12.5 and B20 is comparable to -6 °C [2] and [12] for B20 biodiesel.

Total sulphur content, which is a measure of the corrosive tendencies of fuel, was evaluated. The total Sulphur for diesel, biodiesel-diesel blends and biodiesel samples were determined as shown in Table 4. The full sulphur B0 (petroleum diesel) is 0.137%, higher than B5, B12.5, B20 and B100. However, the total Sulphur in B100 (0.039%) is

well below the recommended minimum value of 0.05% by ASTM D6751 for fuel used as diesel in internal combustion engines [2], while that of B0, B5, B12.5 and B20 are higher. Furthermore, the fuel colour for diesel, biodiesel-diesel blends and biodiesel samples was determined and found within the ASTM D-6751 recommended value of 3 maximums [14]. It was observed that B0, B5, B12.5 and B20 are within recommended colour value except for B100 with the higher colour value of 6. Hence, from the physicochemical properties of the diesel, biodiesel-diesel blends and biodiesel samples presented in Table 4, it can be concluded that B5, B12.5 and B20 are within acceptable limit except for B100 which would require upgrading to meet up with the recommended standard for fuel used as diesel in internal combustion engines.

CONCLUSION

The physiochemical characterization showed that 5, 12.5 and 20 % CTS oil-based biodiesel-diesel blends are within acceptable combustion engine without modification except for 100 % CTS oil-based biodiesel which would require upgrading to meet up with the recommended standard for fuel used as diesel in internal combustion engines. FTIR and GCMS analysis shows the functional groups and characteristic peaks of fatty acids and methyl ester with carbon range from C13–C17 with C16 constituting the major fatty acid group and contains 61.69 % MUFA and 38.31 % SFA with a 94.3 % biodiesel yield.

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