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Biodiesel Production from *Cassia tora* Seed Oil Through Acid-Base Catalysed Method

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ABSTRACT

Biodiesel produced from different triglyceride sources is an alternative fuel to petro-diesel. An investigation was carried out with *Cassia tora* seed oil. The oil was esterified to reduce the FFA content in the oil and trans-esterification of the oil was carried out which involves making use of methanol and sodium hydroxide pellets in a base-catalyzed trans-esterification reaction. 187g *Cassia tora* oil was trans-esterified with 20 g of methanol and 0.7 g of NaOH pellets at 55°C operating temperature. The outcome was a percentage conversion of 90.53 per cent for *Cassia tora* feedstock and a by-product of 15.20 g of glycerol (i.e. soap). The physical features of the oil, such as smell and colour, were retained in the biodiesel produced. The density of biodiesel made from *Cassia tora* oil was determined to be 848.0 kg/m³. The flashpoint was determined to be 135 °C and the acid value and water content were 0.16 (MgKOH/g) and 0.04 (per cent mass), respectively. The kinematic and dynamic viscosities of the generated biodiesel were 4.2mm²/s and 6.49 x 10⁻³kgm⁻¹s⁻¹, respectively. The pour point was also discovered to be -6 °C. The biodiesel was evaluated using American testing and material standards (ASTM Values). Based on the comparison of measured characteristics with ASTM values, *Cassia tora* seed oil could be a promising option for biodiesel production

INTRODUCTION

Because of growing concern about the availability of petroleum-based fuels and the impact of these fuels on the increase in CO₂ levels in the atmosphere, the development of biofuels has lately become a hot subject [1–7]. Increased usage of diesel-generated from fossil oil contributes to air pollution and exacerbates the problem of global warming caused by CO₂ and other particulates [8]. Given its biodegradability, renewability, non-toxicity, low emission of gaseous particle pollutants, and greater cetane number than petrol diesel, biodiesel fuel has gained increasing attention as an alternative fuel. It is also more affordable than petrol diesel [9]. Also important is the fact that it fulfils the world's current rising need for energy and ensures energy security, which is now highly dependent on petroleum-based fuel supplies, which will be exhausted in the near future if the current pattern of energy use is maintained [10].

Due to the fact that biodiesel contains more oxygen than petroleum diesel, the use of biodiesel in diesel engines has resulted in considerable reductions in particle pollution, carbon dioxide emissions, smoke emissions, and noise emissions. Also of note is that the combustion of this vegetable base fuel does not contribute to net atmospheric CO₂ levels and that such fuels are derived from agricultural resources that have been carbon fixed by photosynthetic carbon fixation [11].

The focus of this research is biodiesel production from *Cassia tora* seed oil. *Cassia tora* plant is a large genus classified as a legume under the family leguminosaceae or as a weed in the family caesalpiniaceae. The plant is found enormously in most tropical countries; it is a drought-resistant plant and can grow on a low fertile wasteland [12]. Biodiesel potential of *Cassia tora* was reported [13,14] where it was classified among the twenty-seven potential non-edible biodiesel feedstock.

MATERIALS AND METHOD

Cassia tora oil was used as the substrate in this research work. This was extracted from *Cassia tora* plant obtained from MAUTECH, Yola which was used for the production of biodiesel.

Equipment

Industrial sieve (of 70 μm pore), Beaker, Retort stand, Dropper, Thermometer, Hot plate, 250mL bottom flask, weighing pan, separating funnel, 50 mL and 250 mL conical flask, Magnetic stirrer, Oven, Syringe, Burette, Hand gloves, PH meter, Spatula, weighing balance.

Chemicals/reagents

Methanol, Distilled water, Sodium hydroxide pellets, Potassium hydroxide pellets, Phenolphthalein, Tetraoxosulphate (VI) acid and iso-propanol alcohol.

Testing for free fatty acid (FFA) of the *Cassia tora* oil

In a conical flask, 2g of *Cassia tora* oil was weighed. To dissolve the oil, 25ml of propan-2-ol was applied to the sample. The mixture was then treated with three drops of phenolphthalein. In a conical flask, two more solutions were made using the same process as before, totalling three solutions [15].

Titration

In the burette, 0.1 M KOH was poured and titrated against the mixture of camel foot oil samples. Following a colour change from yellow to pink or a sharp change, the initial and final readings were recorded, followed by the overall average titre value [16].

Removal of water from the oil

Anhydrous chemicals and materials must be employed in all trans-esterification processes. Water in any of the reactants and catalysts will react with biodiesel to produce it (hydrolysis), resulting in the production of free fatty acid (FFA) [17]. The treated *Cassia tora* seed oil (poor in free fatty acids) was baked at 120°C for 20 minutes to allow the water in the oil to evaporate into the atmosphere. Trace beads of soap detected floating on the oil's surface were wiped away [18]. To eliminate any remaining suspended particles, the filtration procedure was repeated. The result was a clear and vivid hue.

Determination of the oil's properties

Some of the features of *Cassia tora* seed oil were determined for usage in the trans-esterification reaction. This will serve as the foundation for the discussion of results.

Determination of the saponification value of *Cassia tora* seed oil

The saponification value is the number of milligrams (mg) of potassium hydroxide (KOH) necessary to saponify the esters and neutralise the free acids in one gram (g) of a sample. It also reveals the average molecular weight of the triglycerides in the oil. In a 250 mL dry round bottom flask, 1 gram of oil was weighed. The oil was treated with 50 mL of 0.5 M alcoholic potassium hydroxide (KOH). The reflux condenser was set up, and the round bottom flask's contents were refluxed for roughly an hour. The mixture was allowed to cool after refluxing before being titrated against standard hydrochloric acid and the titer value was recorded [13].

Similarly, 50 mL of the same alcoholic potassium hydroxide (KOH), blank (no oil added), was refluxed for 1 hour in a round bottom flask, chilled, and titrated against standard 0.5 HCl. The

titer value was measured, and the saponification value will be calculated [19].

Acid-catalyzed stage-1 procedure (esterification)

A weighing balance was used to measure and weigh 200g of *Cassia tora* seed oil in a conical flask. The conical flask containing the *Cassia tora* oil sample was placed on top of a magnetic stirrer and swirled before being heated to 400 degrees Celsius. Immediately after the agitation and heating began, 43.54 mL of methanol and 0.9675 mL of sulfuric acid were added to the oil, and the mixture was continually agitated for 15 minutes, allowing the oil's free fatty acids (FFA) to react. After 30 minutes, two different layers were visible: the top layer was a less viscous waste oil rich in free fatty acids (FFA), while the bottom layer was a soap emulsion. The soap emulsion was separated from the waste [20].

Base catalyzed stage-2 procedure (transesterification)

In the test batch production, 187 gram (g) of *Cassia tora* seed oil and 37.4 gram (g) of methanol (i.e. 20% by volume of oil) were used. A magnetic heater/stirrer was used to pre-heat 187 gram (g) of *Cassia tora* seed oil to a consistent temperature of 60 °C. 20 gram (g) of methanol was measured and poured into the beaker using the measuring cylinder. The weighing scale was used to weigh 0.7 g of NaOH pellet, which was then added to the methanol [21]. The beaker's contents were aggressively swirled with the second magnetic stirrer until the NaOH was entirely dissolved in the methanol. The resulting combination is known as sodium Methoxide. The Methoxide was put into the heating oil-filled conical flask. The magnetic stirrer was used to swirl the contents of the conical flask at a constant speed and temperature of 55 °C. After 2 hours, the heating and stirring were turned off, and the result was poured into a separating funnel 50 set on a clamp stand. From 4:25 p.m. to 10:10 a.m. the next morning, the mixture was allowed to settle. The separating funnel was opened at the bottom to allow the glycerin at the bottom to drain, and the biodiesel was collected in a beaker before being placed into a container for storage [13].

Washing and drying of biodiesel

To eliminate any leftover methanol, catalyst, soap, or other contaminants, biodiesel must be washed [22]. To eliminate all water-soluble contaminants and excess catalysts, 50 mL of distilled water was run through the biodiesel. Water was periodically passed from the top and taken from the bottom of the washing vessel during the washing, and the washing was terminated when a batch of washing water removed was clear enough to demonstrate the purity of the biodiesel. Following washing, the washed biodiesel was gently but steadily heated to 100 degrees Fahrenheit and held at that temperature for 30 minutes, until the majority of the water content was evaporated.

Biodiesel Properties Determination

The following are the several ways for determining the qualities of biodiesel:

Biodiesel Sample Density

The density meter machine was turned on, and the temperature was stabilised. The measuring cell was washed and dried with a little amount of n-Hexane, followed by Acetone. Using a 10 mL plastic syringe, the test sample was then injected into the density metre. It was critical that the filled-in sample be homogeneous and free of gas bubbles. In order to prevent the filled-in sample from leaking, the syringe was left in the filling position [23]. The upper hose was linked for evacuation, while the lower hose was connected for injection. When the reading is stable, the upper-

density value is used. Finally, the biodiesel sample from the density metre was evacuated and flushed with n-Hexane, followed by acetone. Finally, density measurements were taken [24].

Biodiesel Dispensing Point

Biodiesel was poured into the test jar until it reached the level mark, then sealed with the cork containing the high-pour thermometer. The test jar was placed in the pour bath, which was previously adjusted to -150 °C. When the temperature reaches 90 °C above the projected pour point, the appearance of biodiesel was tested at 30 degree intervals. The collected moisture that hinders visibility was removed by wiping the surface with an alcohol-soaked clean towel.

The jar was tilted to see if there was any oil movement. (The entire operation of removal, wiping, and replacement will take no longer than 5 s.) When the oil in the jar did not flow when tilted, the jar was kept horizontally for 5 s and timed with a stopwatch (if the oil shows any movement, the test jar was replaced immediately in the jacket and the test for flow was repeated at the next 3 °C lower). The method was continued until the biodiesel showed no movement when the test jar was held horizontally for 5 s. The observed reading of the test thermometer was recorded as the biodiesel pour point.

Biodiesel's Flash Point

Biodiesel was put into the cup until it reached the flashpoint tester's recommended mark. The flame dipper and flame presentation control arm were both positioned. The chiller bath was turned on and set to room temperature. The electrical panel's analyser was turned on. The intended flashpoint was written and supplied using the front panel's icons and cursors; the method was also selected. The minimum temperature at which biodiesel ignited (flash) when the ignition source was applied was displayed and recorded [25].

Biodiesel Water Content

The water test cell was turned on for the first time. The range interface (0–10% and 0.02–1%) will be displayed on the screen, and the suitable water content range will be selected. And the appropriate approach (Easy SHIP Paste method) was chosen. The cell was opened, and Reagent A (part of the equipment accessories) was vigorously shaken before adding 20ml of the reagent to the cell. The entire contents of an Easy SHIP Paste bottle were added, along with 5ml of the biodiesel sample and an agitator. After replacing the cover, the start button was pressed. The cell was aggressively shaken for 3 minutes until the test was completed. And the outcome was shown at the end of the exam [26].

Kinematic Viscosity of Biodiesel

The viscometer bath was nearly full of pure water, and other gadgets were attached and operated. The setup was left for a while to allow the fixed temperature and warm water to equilibrate. Using a pipette filler, the biodiesel sample was then put into the U tube viscometer. The time required to transport a specific quantity of sample from one side of the U tube to the other was recorded. At the equilibrated temperature, the viscosity of each sample was computed as the product of the average time taken and a constant C (the value of C varies on the type of viscosity metre used) [27].

RESULTS

The result obtained in esterification is shown in **Table 1** The *Cassia tora* seed oil sample was firstly tested for free fatty acid (FFA) by used potassium hydroxide (KOH) with indicator and was found to be 6.45 mg KOH/g which shows that it cannot be used to produced biodiesel because it can cause the formation of soap during trans-esterification. This lead to the esterification process of the oil with methanol and sulphuric acid (H₂SO₄), after esterification the oil was continue been test for free fatty acid using 0.1 M of potassium hydroxide (KOH) as a titrant and subsequently, after six tests, with the same procedure, The process of trans-esterification of *Cassia tora* seed oil gave a yield of 169.30 g biodiesel and 15.20 g glycerol. 40.60 g of the total reacting masses could not be accounted for. Reasons for these losses can be attributed to some alcohol and residual catalyst and emulsions which can be removed during the washing stage of the production process the %FFA was successfully reduced to a value of 0.56% and are presented in **Table 2**. The results stated in **Table 3** are averages of three different experimental runs of the transesterification process. Also, **Table 4** contains the fuel properties of the biodiesel produced and the biodiesel standards mandated by the American system for testing and materials (ASTM values) standard.

Table 1. Titration values.

Burette readings	1 st	2 nd	3 rd	4 th	5 th	6 th
	titration	titration	titration	titration	titration	titration
Final readings (cm ³)	4.60	9.0	5.40	12.60	3.00	0.40
Initial readings (cm ³)	0.00	5.40	2.90	10.20	1.90	0.00
Total volume used (cm ³)	4.60	3.60	2.50	2.4	1.1	0.40

Table 2. Acid value and free fatty acid (FFA) *Cassia tora* seed oil before and after esterification.

Property	Initial value	First ester-ification	Second ester-ification	Third ester-ification	Fourth ester-ification	Fifth ester-ification
Acid Value (g)	12.90	10.24	7.01	6.73	3.09	1.12
FFA (%)	6.45	5.12	3.51	3.36	1.54	0.56

Table 3. Results for the oil Transesterification experiment.

Experimental condition	1st Run	2nd run	3rd run	Average run
ReactionTemperature	55	55	55	55
Reaction time(min)	120	120	120	120
Oil quantity(g)	187	187	187	187
Methanol quantity(mL)	20	20	20	20
NaOH Concentration(g)	0.70	0.70	0.70	0.70
Biodiesel obtainedD	168.45	169.20	170.25	169.30
Glycerol obtained (g)	15.27	15.18	15.15	15.20
Mass losses (g)	22.10	22.40	22.10	22.20
Biodiesel yield	91.05	90.48	91.04	90.53

Table 4. Characterization results for *Cassia tora* biodiesel.

Fuel property	ASTM standard	<i>Cassia tora</i> biodiesel oil
Density(g/cm ³)	0.75-0.84	0.8465
kinematic Viscosity(mm ² /s)	1.9-6.0	4.2
Dynamic viscosity kgm ⁻¹ s ⁻¹		6.49 x 10 ⁻³
Water content(%mass)	0.05 max	0.04
Acid value (MgKoH/g)	0.1	0.16
Flash point (°C)	130min	135
Pour point(°C)	-15 to 10	-6

Table 5. The density of *Cassia tora* oil biodiesel at 25 °C.

Oils	Density at 25 °C (kg/m ³)
Pure oil	935.0
<i>Cassia tora</i> oil	850.8

Table 6. Densities of biodiesel from various vegetable oils at 25 °C.

Oil	Density (Kg/m ³)
linseed	887
canola	825
sunflower	882
rapeseed	877

Table 7. dynamic viscosity of *Cassia tora* and its ester.

Oils	Measured oil at 25 °C (kg/m ³) density
Pure oils	8.42
<i>Cassia tora</i> oils	6.49

DISCUSSIONS

The *Cassia tora* seed oil sample was firstly tested for free fatty acid (FFA) by used potassium hydroxide (KOH) with indicator and was found to be 6.45 mg KOH/g which show that it cannot be used to produce biodiesel because can cause the formation of soap during trans-esterification. This leads to the esterification process of the oil with methanol and sulphuric acid (H₂SO₄), after esterification the oil was continue been test for free fatty acid using 0.1 M of potassium hydroxide (KOH) as a titrant and subsequently, after six tests, with the same procedure, the %FFA was successfully reduced to a value of 0.56%.

The trans-esterification of *Cassia tora* produced 169.30 g biodiesel and 15.20 g glycerol. There were 40.60 g of total responding masses that could not be accounted for. Some of these losses can be ascribed to leftover alcohol, catalyst, and emulsion, which can be eliminated during the washing stage of the manufacturing process. The reported results are the averages of three different experimental runs.

In the associated research of biodiesel ester as a fuel lubricity additive, diesel fuel (DF) was employed as a reference fuel. It should be noted that the density of diesel fuel is affected by the initial crude oil as well as the refining methods used to create the product. The specific gravity of the *Cassia tora* biodiesel was 3.3 per cent greater than the reference (DF).

The densities of the biodiesel produced are comparable to other values (given in **Table 5**) obtained from a similar experiment using different oil sources [28]. It is crucial to remember that the benefits of using *Cassia tora* oil for biodiesel synthesis greatly outweigh the disadvantages associated with their use as feedstocks. The evaluation of biodiesel produced emphasizes the importance of taking both preventive and remedial actions to reduce the discharge of non-environmentally friendly compounds into the environment connected with the disposal of *Cassia tora* and wasted bleaching earth.

In comparison, one study [28] validates the use of *Cassia tora* and waste cooking oils as feedstocks for biodiesel synthesis with the least amount of release of non-environmentally friendly compounds into the environment. The use of these oils (as commercial feedstocks) will help to reduce water and soil pollution caused by unlawful waste oil discharge into rivers and landfills. Every day, massive amounts of these oils are produced

all over the world. The Energy Information Administration estimates 100 million gallons per day in the United States [29].

One of the most common criticisms raised against biofuels, particularly large-scale biodiesel production, is that they may contribute to food shortages and price increases in basic staples. This problem, however, can be overcome by research involving the use of waste oils for the creation of biodiesel rather than virgin oils, and therefore feasible food products with high oil content [30]. One strategy to assure the long-term viability of biodiesel is to use waste oils as feedstocks, such as Crude *Cassia tora*. Furthermore, an increasing human population will always result in a rise in the output of waste cooking oils. Waste cooking oils are critical for low-cost biodiesel manufacturing in order for biodiesel to compete effectively with petroleum diesel (in terms of cost) [2,3,5,31,32]. Because of the low cost of producing biodiesel from waste oils and the ease of creating such feedstocks (waste oils) from a normal household and business activities, it is appropriate for use as an alternative fuel source.

CONCLUSION

Biodiesel manufacture from *Cassia tora* seed oil was carried out, with the FFA content in the *Cassia tora* seed oil being approximately 6.45 per cent. As a result, a two-step acid-pre-treatment was performed to reduce the oil's FFA concentration. This reduced the per cent FFA to 0.56 and allowed for further transesterification activities. The experiment yielded a 90.53 per cent conversion rate of *Cassia tora* to biodiesel. *Cassia tora* product density was determined to be 848.0kgm⁻³. This value is equivalent to values obtained from a similar experiment using different oil sources. *Cassia tora* bio-diesel has kinematic and dynamic viscosities of 4.2 mm²/s and 6.49 x 10⁻³ kgm⁻¹s⁻¹, respectively. The resulting biodiesel kept the original feed stock's size and colour. At 25 °C, the viscosity of the *Cassia tora* biodiesel was reduced by 85.2 per cent compared to the raw vegetable oils. The limited fuel characterization performed revealed that the *Cassia tora* biodiesel produced may successfully power a diesel engine.

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