

RESEARCH ARTICLE

EGGSHELL HETEROGENEOUS CATALYZED IN-SITU TRANSESTERIFICATION OF CASSIA TORA SEED (CTS) OIL

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ABSTRACT

Diminishing fossil fuel resources, coupled with the steady increase in energy consumption has spurred research interest in alternative and renewable energy sources. The article presents the production of biodiesel from cassia tora seed using eggshell as catalyst. In this research work, oil was extracted from CTS using soxhlet extractor and n-hexane as solvent. The extracted oil was characterized via physicochemical analysis then in-situ transesterification of oil with methanol was carried out using eggshell as catalyst. The physico-chemical analysis showed the state of the oil at room temperature to be liquid, the odour was mild, the colour was brown, specific density was 3.46, saponification value was 193.6 ± 0.25, acid value was 8.2 ± 0.4, and iodine value was 100.68 ± 1.0. Biodiesel characterization showed that kinematic viscosity at 40°C was 4.98, cetane number was 64.5, flash point was 159, pour point was -13, cloud point was 5, and density was 0.85. The prepared eggshell catalyst was characterized using X-ray Fluorescence and was found to be composed of CaO (73.2), MgO (0.2), SiO₂ (0.4), SO₃ (0.3), P₂O₅ (0.7), Na₂O (0.5), Al₂O₃ (0.07), K₂O (0.03), and Fe₂O₃ (0.08). This study suggests that CTS oil can be used as a source of triglycerides in the manufacture of biodiesel by transesterification. The properties of the biodiesel produced showed theoretical compatibility with diesel engines in accordance with the American standard for testing materials (ASTM).

KEYWORDS

Biodiesel, Transesterification, Cassia Tora Seed, Egg shell.

1. INTRODUCTION

The world energy sector depends on petroleum, coal, and natural gas reservoirs to fulfill its energy requirements (Meher et al., 2006). Nigeria is traditionally an energy deficient country which exports above 70% of its crude oil production. The country is dependent upon import of petroleum products to sustain its growth. Diesel fuel plays an essential function in the industrial economy of Nigeria. The fuel is used in heavy trucks, city transport buses, electric generators, farm equipment etc. (Anjana, 2000). However, diesel engine also emits various forms of pollutants into the environment which can endanger human health and damage the ecological environment (Antolin et al., 2002). It is therefore essential that the world extend its interest towards new sources of energy. A relatively new alternative that is currently booming worldwide is fuel obtained from renewable resources or biofuel. Biofuels are well suited for decentralized development i.e. it can be utilized to meet the need for social and economic progress, especially in rural communities where fossil fuels may be difficult or expensive to obtain (Ezeanyanaso et al., 2010). Amongst the various alternative fuels which could match the combustion features of diesel oil and can be easily adapted for use in existing engine technologies with or without any major modifications is biodiesel. Biodiesel fuel produced from vegetable oils (both edible and non-edible) or animal fats is one of the promising possible sources that can be substituted for conventional diesel fuel and produces favourable effects on the environment. Biodiesel is recommended for use as a substitute for petroleum diesel mainly because it is a renewable, domestic resource with

an environmentally friendly emission profile and is readily available and biodegradable. The research and use of biodiesel fuel as an alternative started in the 1980's and the reason was the diesel crisis caused by the reduction of petroleum production by the Organization of Petroleum Exporting Countries (OPEC) and the resultant price hike. The biodiesel produced from locally available resources offers a great promise for future application in Nigeria as it can help in attaining much needed energy security and being environment friendly, will help to conform to stricter emission norms (Carraretto et al., 2004).

For the purpose of this study, CTS oil was extracted from the kernels of the Cassia Tora tree of the Eabaceae family. The plant is considered a weed in many places which occurs through west, north-east and east tropical Africa across a range of vegetation types, principally wooden grassland and dry savannah of the northern tropical Africa and Sahel region, and this include northern Nigeria. There are more than 350 oil bearing crops identified, among which are sunflower, safflower, soybean, castor seed, Jatropha seed, cottonseed, rapeseed and peanut oils, which are considered as potential feedstock for biodiesel production.

The use of vegetable oil for biodiesel production may result in increase in price of food or lead to food shortages (Ezeanyanaso et al., 2010). Fortunately, non-edible vegetable oils, mostly produced by seed-bearing trees and shrubs can be alternative without competing food uses. Crude edible oils however have high free fatty acid (FFA) content, which affects biodiesel yield and capital cost. These oils can be pre-refined by reducing the free fatty acid content (FFA) using esterification and saponification

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processes (Wang et al., 2006). The fuel properties of biodiesel differ from those of petro-diesel fuels. This implies that different engine performance and emissions will occur when biodiesel is used in diesel engines (Carraretto et al., 2004). Compared to petroleum based diesel, the high cost of biodiesel is a major barrier to its commercialization; its cost is 150% more than that of petroleum-based diesel depending on feedstock used. Approximately 70-95% of the total biodiesel production cost arises from the cost of feedstock, solvent and refining process of the crude oil (Hass et al., 2006). Previous work on biodiesel has not quantified the biodegradability of CTS oil biodiesel. Furthermore, work is still ongoing by scientists on better methods of optimization of biodiesel.

Energy is the main driver of socio-economic growth of any nation. It plays a vital role in the overall framework of development worldwide. Energy is an indispensable commodity and all aspect of human activities is hinged to it, it is also as a factor of production whose cost directly affects price of other goods and services (Dermibas, 2005). Access to energy has been described as a key factor in industrial development and in providing vital services that improve the quality of life as well as the engine of economic progress (Singh et al., 2004). Diesel fuels produced from vegetable oils have practically lesser sulphur content, lesser greenhouse gases emissions especially with CO₂, offer no storage difficulty, and they have excellent lubrication properties. Moreover, vegetable oils, yielding trees absorb more carbon dioxide from the atmosphere on burning (Erhan et al., 2006). Hence, diesel fuels essentially help to alleviate the increasing carbon dioxide content in the atmosphere. With this development, there has been a renewed focus on vegetable oils to make biodiesel fuels. The substitution of diesel oil by renewable fuels produced within the country would generate higher foreign exchange savings, even for the major oil exporting countries like Nigeria. The Cassia Toraleaves and roots in many countries for decades has been used for treatment of malaria, yellow fever and typhoid but the potential of CTS oil has not been properly explored. (Jain et al., 2010).

In this present research work, biodiesel was produced from Cassia tora seed using eggshell as a catalyst. This in turn reduces the effect of environmental issues. Enough investigations are seen in the literature for biodiesel production, and no study is seen for production of biodiesel from cassia tora seed using eggshell as catalyst and is considered for evaluation in the present study. Thus, the aim of this research is to study the application of eggshell Heterogeneous Catalyst in Transesterification of Cassia Tora Seed (CTS) Oil.

2. MATERIALS AND METHOD

2.1 Sample Collection and Treatment

The plant seeds of Cassia Tora were gotten from Aleiro local government area of Kebbi State on the 15th of February 2021. The seeds collected were properly sun dried to enhance the removal of the seed coat; its inclusion will make extraction less efficient. The dehulled seeds were further dried at room temperature for days. The dried seeds were crushed into cake using mortar and pestle in order to weaken the cell walls to release oil via extraction.



Figure 1: Raw Cassia Tora seeds

2.2 Oil Extraction

150 ml of normal hexane was poured into round bottom flask. 100 g of the sample was placed in the thimble and was inserted in the centre of the extractor. The Soxhlet was heated at 65 °C. When the solvent was boiling, the vapour rose through the vertical tube into the condenser at the top. The liquid condensate dripped into the thimble in the centre, which contained the solid sample to be extracted. The extract seeped through the pores of the thimble and filled the siphon tube, where it flowed downward into the round bottom flask. This process continued for a period of 4-6 hours. It was then removed from the tube, dried in the oven, cooled in the

desiccators and weighed again to determine the amount of oil extracted. Further extraction was carried out. The extracted oil was stored in a container for subsequent characterization.

2.3 Physico-chemical analysis

2.3.1 Determination of iodine Value

About 0.25 g of the oil sample in a 250 mL conical flask was added 10 mL of chloroform followed by 30 mL of Hanus iodine solution. The flask was securely closed and the solution was kept in the dark after shaking for 30 minutes. Then 10 mL of 15% potassium iodide solution was added and shaken. To the resulting mixture 100 mL of distilled water was added. The mixture was then titrated with the iodine solution against 0.1 M Sodium thiosulphate solution till a yellow color formed. This was followed by addition of 2 drops of starch solution after which a blue solution was formed. The titration continued until the blue colour disappeared while the volume of

Na₂S₂O₃ at end point was recorded. The Iodine value (I.V) was calculated using equation (1) (Al-Hamdany et al., 2012).

$$I.V = \frac{12.69 \times C \times (V_1 - V_2)}{m} \quad (1)$$

Where C= Concentration of Na₂S₂O₃ used; V₁ = volume of Na₂S₂O₃ used for the blank; V₂ = volume of Na₂S₂O₃ used for sample; m = mass of the sample.

2.3.2 Determination of saponification Value

About 2 g sample of the oil was weighed into a 250 mL glass conical flask, and then 10 mL of ethanol ether mixture (2:1) was added to the same flask followed by 25 mL of 0.5 N ethanolic potassium hydroxide. The flask was then fitted to a reflux condenser and refluxed using a boiling water bath for 30 min with occasional shaking. To the warm solution, 3 drops of phenolphthalein indicator were added and the warm solution was titrated against 0.5 M HCl to the end point. The same procedure was used for other runs and blank. The expression for saponification value (S.V) is calculation using equation (2) (Kyari, 2008).

$$S.V = \frac{56.1 \times M \times (B - R)}{m} \quad (2)$$

Where B = the volume of the solution used for blank test; R= the volume of the solution used for determination;

M = Molarity of the HCl used; m = Mass of the sample.

2.3.3 Determination of acid Value

Into a dried 250 mL conical flask, 2.0 g of the oil sample was placed followed by 25 mL of absolute ethanol and 3 drops of phenolphthalein indicator. The mixture was heated in a shaking water bath for 5 minutes. While hot, it was titrated against 0.1 M KOH until pink colour appeared. Vigorous shaking was done when approaching the end point to ensure thorough mixing. The volume of 0.1 M KOH consumed by an acid was recorded. The acid value was calculated as reported using equation (3) (Kyari, 2008).

$$A.V = \frac{56.1 \times V \times M}{m} \quad (3)$$

Where V = volume of KOH used; M = molarity of KOH and m = mass of sample.

2.3.4 Determination of iodine Value

To 0.25 g of the oil sample in a 250 mL conical flask was added 10 mL of chloroform followed by 30 mL of Hanus iodine solution. The flask was securely closed and the solution was kept in the dark after shaking for 30 minutes. Then 10 mL of 15% potassium iodide solution was added and shaken. To the resulting mixture 100 mL of distilled water was added. The mixture was then titrated with the iodine solution against 0.1 M Sodium thiosulphate solution till a yellow color formed. This was followed by addition of 2 drops of starch solution after which a blue solution was formed. The titration continued until the blue colour disappeared while the volume of

Na₂S₂O₃ at end point was recorded. The Iodine value (I.V) was calculated using equation (4) (Al-Hamdany et al., 2012).

$$I.V = \frac{12.69 \times C \times (V_1 - V_2)}{m} \quad (4)$$

Where C= Concentration of $\text{Na}_2\text{S}_2\text{O}_3$ used; V_1 = volume of $\text{Na}_2\text{S}_2\text{O}_3$ used for the blank; V_2 = volume of $\text{Na}_2\text{S}_2\text{O}_3$ used for sample; m = mass of the sample.

2.3.5 Determination of Density

Density depends on fatty acid composition and minor components as well as on the temperature [14]. If an oil has low density, it indicates that it contains low molecular weight fatty acids, likewise it will have high saponification value, which means that it can be used for soap production (Afolabi, 2008).

About 25ml of the oil was measured in a pre-weighed measuring cylinder. The weight of the cylinder and the oil was taken; the weight of the oil was obtained by subtracting the weight of the cylinder from the weight of the oil and cylinder. The specific density of the oil is obtained using the equation (3) below (John, 2008).

$$\text{Density of the oil} = \frac{W_1 - W_0}{V_0} \quad (5)$$

Where W_1 = weight of empty measuring cylinder + oil,

W_0 = weight of measuring cylinder,

V_0 = volume of oil

2.4 Catalyst Preparation (Eggshell)

Eggshell which is mainly composed of CaCO_3 was converted into calcium oxide (CaO) by calcination at 800°C for 24hrs (Boro et al., 2011). The eggshell was washed with distilled water to remove impurities, followed by drying in an oven at 105°C . The eggshell was then crushed using a mortar to fine particles. The fine particles were calcined in a muffle furnace at 800°C to eliminate any form of carbon and get a complete conversion of CaCO_3 to CaO. The ash obtained was used as a heterogeneous base catalyst in the production of methyl ester.

2.5 Catalyst Characterization

Different element compositions and components of the catalyst is determined using X-ray fluorescence (XRF) (Thermo scientific ARL OP-TIMX 166).

2.6 Transesterification Process

About 100ml of oil was taken into a beaker, then 20ml of methanol was measured into a separate beaker and 1g of CaO which is the catalyst was dissolved in it. The solution was then poured into the beaker containing the oil. This was then heated in a water bath at 60°C for 1hr, after which it was allowed to settle for 24hrs. Two layers appeared. It was then centrifuged for an hour and finally the biodiesel was obtained (Zhang et al., 2003). Glycerol is heavier than biodiesel therefore it will settle down at the bottom due to gravity. Separating funnel will be used to drain the glycerol from biodiesel.

Water soluble methanol and glycerol was removed by washing intensively with water. The biodiesel produced was filtered to remove the catalyst and residual methanol before vacuum evaporation.

The biodiesel thus obtained was further analyzed to investigate its properties so as to be sure that the produced material would be able to serve its purpose.

$$\% \text{ Biodiesel yield} = \frac{\text{weight of biodiesel produced}}{\text{weight of oil used}} \times 100 \quad (6)$$

2.7 Determination of Biodiesel Fuel Properties

2.7.1 Kinematic viscosity at 40°C (ASTM D 445-06)

A viscometer was inserted into a water bath with a set temperature and left for 30 minutes. The sample was added to the viscometer and allowed to remain in the bath as long as it reaches the test thermometer. The sample was allowed to flow freely and the time required for the meniscus to pass from the first to the second timing mark was taken using a stop watch. The procedure was repeated a number of times and the average value were taken which was then multiplied with the viscometer calibration to give the kinematic viscosity (ASTM D445-06, 2006).

2.7.2 Flash point (ASTM D 93-08)

A sample of the biodiesel was heated in close vessel and ignited. When the sample burns, the temperature was recorded; the pensky-martens cup

tester measures the lowest temperature at which application of the test flame causes the vapor above the sample to ignite. The biodiesel was then placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover was then fitted onto the position on the cup and Bunsen burner was used to supply heat to the apparatus at a rate of about 5°C per minute. During heating, the oil was constantly stirred. As the oil approaches its flashing, the injector burner was lighted and injected into the oil container after every 12 seconds interval until a distinct flash was observed within the container. The temperature at which the flash occurs was recorded, it was repeated three times and the average was taken (ASTM D93-08, 2008).

2.7.3 Pour Point (ASTM D 97-11)

A sample of the biodiesel was kept in the freezer to about 500°C , and then placed in a heating mantle to melt. The temperature at the bottom of the test jar that is the temperature at which the biodiesel starts to pour was taken as the pour point (ASTM D97-11, 2011).

2.7.4 Density (ASTM D1298-99)

A clean dry empty 25ml measuring cylinder was weighed and the mass was recorded as M_0 , it was then filled up with biodiesel. The mass of the bottle and biodiesel was taken and recorded as M and hence, the density was evaluated using the equation below (ASTM D1298-99, 1999).

$$\text{Density} = \frac{M - M_0}{V} \quad (7)$$

2.7.5 Cetane Number (ASTM D 613-17b)

Cetane Number is a measure of the fuels ignition delay. Higher cetane numbers indicate shorter times between the injection of the fuel and its ignition. (ASTM D613-17b, 2017) It was calculated using the equation below

$$\text{CN} = 4.63 \pm \frac{5458}{SV} - 0.2251V \quad (8)$$

3. RESULTS AND DISCUSSION

In table 1, the results of physicochemical analysis of the Cassia Tora seed oil is given. Iodine value obtained for CTS oil was found to be $100.681 \text{ I}_2/100\text{g}$ and it falls within the AOCS limit. The iodine value shows the level of unsaturation of the oil and also influences the oxidation and deposition formed in diesel engines. It is used in determining the drying property of the oil. It is classified as non-drying oil since the iodine value is below $115 \text{ gI}_2/100\text{g}$ (Abdulkareem et al., 2011). The saponification value obtained for CTS oil was found to be 193.6 mg KOH/g and it is within the range specified by AOCS. The saponification value of oil is a measure of the tendency of the oil to form soap during the transesterification reaction. This high value indicates that it has a high tendency of forming soap during transesterification reaction. The acid value obtained for CTS oil was found to be 8.2 mg KOH/g . The value exceeds the AOCS limit of 2.0 mg KOH/g . A recent study reported that oils with high acid value tend to deactivate catalyst used during transesterification (Kyari, 2008).

Table 1: Results of physico-chemical properties of the CTS oil.

Parameters	Value
Colour	brownish
Odour	mild
State at room temperature	liquid
Density (mg/g)	3.46
Acid value (mg KOH/g)	8.2 ± 0.4
Saponification value (mg KOH/g)	193.6 ± 0.25
Iodine value (mg iodine/g)	100.68 ± 1.0

In table 2, the results of fuel characterization of the biodiesel obtained from CTS oil are given. The biodiesel produced was characterized using ASTM specification methods. The table contains some of the characterized properties of the biodiesel in comparison with different ASTM specifications and other literatures. These parameters include the following:

3.1 Kinematic Viscosity at 40°C

Kinematic viscosity is a measure of resistance of fluid flow under the influence of gravity (Gerpen et al., 2004). The result of this work (4.98

mm/s²) is in agreement or falls within the ASTM range of 1.9 – 6.0 mm/s². Viscosity of a fuel is related to the lubricity. Low viscosity fuels are unlikely to provide satisfactory lubrication in fuel injection pumps; these often lead to seepage and increase wear (Raja et al., 2011). High viscosity in fuel are responsible for atomization of fuel, incomplete combustion and increased exhaust emissions, choking of the injections thereby forming larger droplets on injectors, ring carbonization and accumulations of the fuel in the engine. Having the value of viscosity neither lower nor higher the ASTM range, it indicates that the produced biodiesel is safe for use in diesel engines (Wang et al., 2006).

3.2 Flash point

Flash point is the minimum temperature at which a fuel must be heated for it to ignite air-vapor mixture. The U.S department of Transportation specified 90°C as the flash point for non- hazardous fuel (Kenneth et al., 2010). The result of this work (15°C) shows appreciable consistency with ASTM specification of (>130°C) and it shows a lower value when compared to other research works who reported a value of (163°C) for waste cooking oil (Ayoola et al., 2012). Other study reported a value of (158°C) for waste cooking oil (Hillary and Christopher, 2013). The high value obtained in this study clearly signifies that the biodiesel produced is basically free from methanol; this is because even small quantity of methanol can reduce the flash point reasonably and also negatively affect diesel engine parts such as fuel pumps, seals and elastomers (Wang et al., 2006).

3.3 Cetane number

Cetane number is a measure of ignition quality of diesel fuel. The higher the cetane number, the easier the fuel will ignite when it is injected into the engine, the better the fuel (Kenneth et al., 2010). Beside the reduction of viscosity resulting from transesterification of vegetable oil, one of the most evident changes that result from process is the significant increase in cetane number of the fuel produced. The result of this work (64.5) is in agreement or falls within the ASTM specification (47-65). This result is higher when compared with who reported a value of (49.2) for sunflower (Mansourpour and Shariati, 2012), and others reported a value of (48.63) for microalgae oil (Misau et al., 2015) and reported a value of (47) for *Azadirachta indica* oil (Muthu et al., 2010).

3.4 Pour point

Pour point is a criteria used for low temperature performance of a fuel (Alamu et al., 2007). The result of this work (-13°C) is in agreement or falls within the ASTM range of -15 to -10°C. The low temperature obtained indicates the biodiesel can still flow in a very low temperature.

3.5 Cloud point

Cloud point has implication on the use of biodiesel in cold weather. The cloud point is the most common measure of tendency of a fuel to crystallize and the cloud point in this work was found to be 5°C. This signifies that *Cassia tora* oil has a high tendency of forming cloudy crystals easily in cold temperature.

Parameters	Values	ASTM
Kinematic viscosity at 40°C	4.98	1.9-6.0 ASTM D 445-06
Cetane number	64.5	47-65 ASTM D 613-17b
Flash point (°C)	150	>130 ASTM D 93-08
Pour point (°C)	-13	(-15) – 10 ASTM D 97-11
Cloud point (°C)	5	3-12
Density (g/cm ³)	0.85	0.845 ASTM D 1298-99

In table 3, the results of chemical composition of the catalyst eggshell on dry basis obtained using XRF is given. The compounds identified in the catalyst include CaO, MgO, SiO₂, SO₃, P₂O₅, Na₂O, Al₂O₃, K₂O, and Fe₂O₃. Some of these compounds are basic, others are acidic and one of them is amphoteric in nature. The value obtained in wt % for these compounds indicated that CaO is the principal compound in the eggshell catalyst which is a basic compound and therefore can be used as alkaline catalyst for transesterification of the CTS oil for biodiesel production.

Major oxide	wt (%)	Nature
CaO	73.2	Basic
MgO	0.2	Basic
SiO ₂	0.4	Acidic
SO ₃	0.3	Acidic
P ₂ O ₅	0.7	Acidic
Na ₂ O	0.5	Basic
Al ₂ O ₃	0.07	Amphoteric
K ₂ O	0.03	Basic
Fe ₂ O ₃	0.08	Basic

4. CONCLUSIONS

Egg shell Heterogeneous Catalyzed in-situ Transesterification of Cassia Tora Seed (CTS) Oil was successfully used in converting desert date seed oil to biodiesel. The properties of the biodiesel produced showed theoretical compatibility with diesel engines in accordance with the ASTM. Thus, eggshell catalyzed in situ transesterification was found to be a suitable method for conversion of CTS oil with high saponification value into biodiesel. Finally, the study shows that the biodiesel from seeds of Cassia tora plant is environmentally friendlier than petroleum diesel. Also, the high saponification value obtained for Cassia Tora seed oil shows that it can also be used in the manufacture of soap.

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