

Research Article

# Physicochemical Characterization and Production of Biodiesel from Cottonseed Oil and Waste Cooking Oil

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## Abstract

Biodiesel is an eco-friendly, alternative diesel fuel prepared from domestic renewable resources i.e. mainly produced from vegetable oils and animal fats. It is a renewable energy source that appears to be the perfect answer to the world's energy needs, especially those of Ethiopia. The aim of present study was to evaluate the physicochemical characterization and production of biodiesel from cottonseed oil and waste cooking oil. Transesterification of edible and non-edible oil with methanol in the presence of strong acid or base catalysts is the standard process for creating biodiesel. The percent yield of cottonseed crude oil was found to be 62.98 % upon the extraction from the cotton seeds. After food residues and sediments were removed using chemical coagulation with 2%  $\text{Al}_2\text{SO}_4$  as a coagulating agent and gravitational sedimentation, approximately 90.24 percent of the oil was recovered. The physicochemical parameters of oils and its biodiesel were performed and the experimental results such as moisture content (0.32% and 0.27%), specific gravity (0.86-0.9258), viscosity (4.1-65 $\text{mm}^2/\text{sec}$ ), saponification value (56.1-182.3 mg/g), Iodine value (51.74-120.53  $\text{mgI}_2/\text{g}$ ), Acid value (0.30-0.50 mg/g), free fatty acid content (0.23-1.9%), cetane number (74.6-137.56) and higher heat values (40.87-48.94 MJ/kg) are good agreement with ASTM standards. In conclusion, the result of recent study confirmed that the cottonseed oil and waste cooking oil derived biodiesel is an alternative renewable biofuel for petro-diesel with an eco-friendly.

## Keywords

Biodiesel, Cottonseed Oil, Saponification, Transesterification, Waste Cooking Oil

## 1. Introduction

Due to the depletion of oil sources, greenhouse gas emissions causing climate change, and the need to support domestic rural economies, interest in the use of biofuels has increased significantly on a global scale in recent years. [1, 2]. A vegetable oil or animal fat is chemically reacted with an

alcohol, such as methanol, to create biodiesel, an alternative fuel for diesel engines. [3]. Globally; the transportation sector is a major source of energy consumption and carbon dioxide ( $\text{CO}_2$ ) emissions. This sector is responsible for 18% of primary energy consumption and 23% of all energy-related  $\text{CO}_2$

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emissions globally. The global energy crisis and environmental concerns have sparked research on renewable energy sources, with biodiesel, derived from vegetable and waste oils, promising for eco-friendliness. [4].

Many academics have been looking for an efficient fuel alternative in recent years due to the depletion of global crude oil reserves and the worsening climatic conditions linked to the use of fossil fuels. Biodiesel is one of the most promising alternative fuels and has gained international attention. This study examines the physicochemical properties of cottonseed oil and waste cooking oil, evaluating their use as biodiesel feedstock, identifying optimal processing conditions, and comparing performance to conventional diesel. [5].

Vegetable and seed oils are increasingly being used globally as industrial raw materials and for residential purposes (cooking oil). Oils are used by the pharmaceutical industry as a raw material or as an ingredient for making drugs. While the cosmetics industry uses oils as raw materials for various goods, oils are also employed as raw materials in the creation of paint. These various uses for oils encourage the pursuit of high-quality vegetable and seed oils to satisfy the growing demand for them globally. [6-10]. Amongst biofuels, biodiesel is considered the best choice in the face of environmental pollution, the decreases in crude oil reserves followed by exponential price growth. Biodiesel is safer in handling and in storing as a fuel, because its flash point is more than petroleum based fuel. Because the cost of different biodiesel feedstocks varies depending on availability and production technique, biodiesel production offers a substitute for the manufacture of clean, biodegradable, non-toxic, and renewable fuels that are in high demand globally. [11, 12].

The biodiesel combustion by products is better not only for inhabitants but also for earth's environment. Compared to traditional diesel fuel, the emissions of unburned hydrocarbons, carbon monoxide, and specific matter from burning biodiesel are significantly lower. [13, 14]. As biodiesel is produced from natural sources, it contains very few amount of sulfur which leads to less emissions of sulfur dioxide when it burns in an engine [14]. Biodiesel is environment friendly liquid fluid similar to conventional diesel fuel in engine tests, the power and fuel consumption [16-18]. A mono alkyl ester of long-chain fatty acids (methyl, ethyl, or propyl), biodiesel is mostly produced via alcoholysis of tri-alkyl glycerides (TAG) of vegetable or animal fats, such as peanut, cottonseed, soybean, or palm oil. This study highlights the significant role of waste cooking oil in promoting circular economy principles, exploring sustainable energy resources, enhancing energy security, creating rural economic opportunities, and reducing greenhouse gas emissions. [19, 20].

It is primarily made by a transesterification reaction with alcohol and feedstock in the presence of a catalyst. (methanol, ethanol or enzyme) [21]. Identifying the physicochemical properties of cotton seed oil and waste cooking oil, determining how they influence the productivity and quality of biodiesel produced through transesterification are basic questions of this study. The choice of catalyst for alcoholysis reaction depends on the free fatty acid (FFA) content of the

feedstock. Usually, base catalysts such as NaOH, KOH,  $\text{NaOCH}_3$  and  $\text{CH}_3\text{OK}$  are more effective than acid catalysts, but their tendency of generating soaps when the FFA of the feed oil is higher than 3, which lead to difficulties in the downstream purification of the product [22].

## 2. Materials and Methods

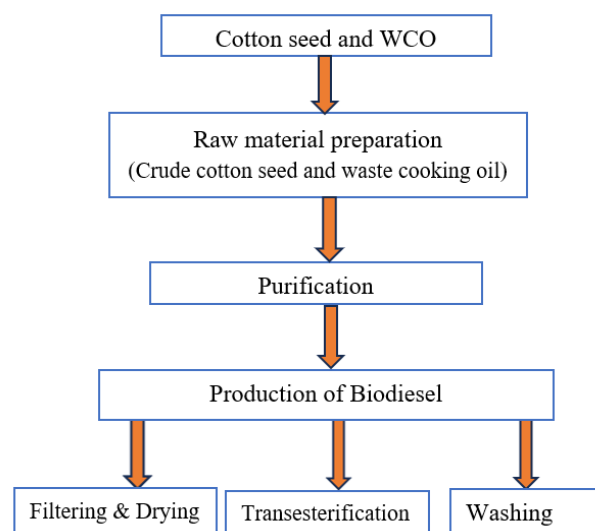
### 2.1. Materials

The raw Material (*i.e.* Cotton seed and WCO) were Collected from the cafeterias (WCO) were located in Located in Chamokebele, ArbaMinch, and purchased (Cotton seed) from Arbaminch market, Ethiopia. Potassium hydroxide (88% pure) pellet, 99% pure methanol, concentrated sulfuric acid (95% pure), phenolphthalein indicator, and sodium sulfate were employed in this study.

### 2.2. Synthesis

#### 2.2.1. Raw Material Preparation

A step-by-step procedure in Biodiesel production is presented in Figure 1. The CS was manually cleaned and preserved in plastic container to avoid the contamination of any foreign matter. The used oil sample was taken from the fryer which was used for frying potatoes and other vegetables based on food items. The temperature observed during frying was in the range of 130 °C-175 °C.



**Figure 1.** Schematic flow chart showing the production of biodiesel from Cotton seed and WCO.

#### 2.2.2. Biodiesel Production Process Flow

This is the primary chemical reaction for biodiesel Production, where triglycerides in the oils react with an alcohol. This involves mixing, heating, high heat reaction time and

separation. The complete flow chart of experimental procedures is provided in Figure 2.

### 2.3. Collection of Samples

Waste cooking oil (WCO) was collected (under convenient sampling method) from the cafeterias were Located in Chamo kebele, Arba Minch. The used oil sample was taken from the fryer which was used for frying potatoes and other vegetables based food items. The Temperature observed during frying was in the range of 130 °C-175 °C. This Temperature could be comparable with the temperature around 140-180 °C for preparing French fries. The collected WCO was stored in plastic container in laboratory for further processing. A Cotton seed (shown in Figure 1) was purchased from Arbaminch market, SNNPR, Ethiopia as the primary raw materials used in production of biodiesel are cottonseed oil. The collected seeds were inspected, manually cleaned and preserved in plastic container to avoid the contamination of any foreign matter. These are used as raw material for the production of biodiesel. Also, these materials contain triglycerides, free fatty acids, and other contaminants.



Figure 2. Cotton Seed collected from market.

### 2.4. Preparation of Cotton Seed for Oil Extraction

The Cleaned cotton seeds were sun dried in open atmosphere until the casing splits and sheds the seeds. The seeds were further dried for about 7 hours (at 60 °C) in hot air-oven to achieve a constant weight in order to reduce its moisture content, which was initially at  $\approx 5$  to 7%. The separation of the shell from the nibs (cotyledon) was carried out using tray to blow away the cover in order to achieve very high yield. Mortar and pestle was used to crush the seeds into a paste (cake) in order to weaken or rupture the cell walls that to release the fat for extraction.

### 2.5. Purification of WCO

The stored WCO was purified by sedimentation and coagulation. The WCO was taken into sedimentation tube that was carry out until sediments were reside at bottom of the tube under gravitational sedimentation method, sediments were

removed by filtration. Further, WCO sample was clarified under coagulation by addition of 2%  $\text{Al}_2\text{SO}_4$  by employing mechanical shaker with a constant agitation at 800 rpm. Clear oil was obtained that was used for the production of biodiesel.

### 2.6. Extraction and Purification of Cotton Seed Oil (CSO)

Oil content in cottonseed samples was extracted and determined using Soxhlet extraction method (see Figure 2). Grounded cottonseed kernels (35 g) was weighed and taken into 50 mm x 125 mm paper thimble and was covered with a piece of cotton before placed it in the Soxhlet extractor. About 350ml of commercial hexane (40% n-hexane) was taken in round bottom flask, and it was used as a Solvent for the extraction of oil. The extraction was continued by heating at 60 °C for 2 hours. The crude cottonseed oil (shown in Figure 3) was obtained and isolated from n-hexane by employing rotary evaporator.

The amount of oil extracted was then measured by weighing the sample after it had been taken out of the evaporator, dried, and allowed to cool to room temperature before being placed on desiccators. The amount of oil extracted by Soxhlet was considered to be the total amount of oil available in the sample.

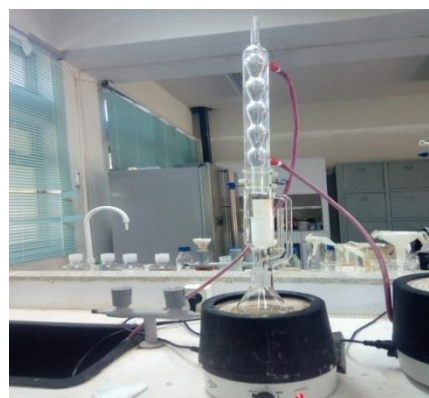


Figure 3. Experimental Soxlet extractor setup.



Figure 4. Extracted crude cottonseed oil.

About 100 ml of water was taken into a separating flask and heated until temperature rises to 70 °C. Obtained CSO was added into the separating flask containing hot water, and wait for 30 minutes until impure particles settles down. Then, hot water containing CSO was transferred into another separating funnel and shaken well vigorously for 5-10 minutes. Because of low density of CSO settles on top of the funnel and high density water and impurities settled at bottom of the separating flask. Then disperse water and impure particles by opening the funnel valve, after that the purified CSO was collected into the vessel. The purified CSO was heated to 65 °C to remove moisture contained in oil [1].

## 2.7. Production of Biodiesel by Transesterification

The steps involve the production of biodiesel from CSO and WCOs:

### Step 1: Drying Filtering and Drying

Food particles that could clog fuel lines must be removed from the spent cooking oil by filtering it. This was carried out while the sample was being prepared. Since water decreases transesterification effectiveness, the oil was dried as much as possible.

### Step2: Transesterification process

Base-Catalyzed transesterification method was adopted for the preparation of Biodiesel. Accurately measured volume (100 ml) of oil sample was taken in 250 ml three-necked round bottom flask (Figure 4). The Oil was preheated to 50 °C by using water bath with temperature regulator. About 1.5g of catalyst potassium hydroxide (KOH) was dissolved in 20 ml of methanol. The flask containing the heated oil was filled with the potassium methoxide solution. A hot plate magnetic stirrer set to 650C and 500 rpm was used to support the experimental setup. To finish the transesterification, the appropriate alcohol-hydroxide solution with vegetable oil was heated for roughly 2 hours. Then, 50 ml of water was measured and poured gently on the product sample to purify it. The mixture was left overnight to properly settle the two phases the biodiesel phase and the water-impurity phase after being gently agitated to prevent foam formation. [2]. A separating funnel was used to separate the product from the glycerol. The resulting mixture is transferred to the separating funnel that holds the glycerine and biodiesel. After 24 hours, glycerin was observed and settles down at bottom of the funnel and biodiesel settles on top of separating funnel. As seen in Figure 4, glycerin was separated by opening the separating funnel's valve.

### Step 3: Washing

To get rid of the soapy deposits and remaining methanol (about a tenth by volume), the biodiesel's methyl ester must be rinsed once more. By settling, the glycerol separates into its own layer. Selling the glycerol to soap and cosmetics producers for purification into pharmaceutical-grade glycerol ought to be feasible. As an alternative, it might be useful in nearby soap manufacturing companies. Due to the presence of

colors from the waste oil, the glycerin is now colored; this will be eliminated commercially by filtering it through charcoal. The separated biodiesel was Obtained (shown in Figure 6) and it was then heated to 100 °C for 1 hour to evaporate the remaining water molecules on it. By comparing the weight of layer biodiesel with the weight of CSO/WCO, the percentage of the biodiesel yield was calculated. [1, 23].



Figure 5. Transesterification experimental set up.



Figure 6. Biodiesel separation.



Figure 7. Purified biodiesel.



$$\text{Biodiesel yield (\%)} = \frac{\text{Grams of methyl esters produced}}{\text{Grams of oil used in reaction}} \times 100$$

## 2.8. Physicochemical Characterization of Oil and Biodiesel

Moisture content, saponification value, acid value, iodine value, free fatty acid content, ester value, specific gravity, higher heating value, viscosity, and cetane number were among the physicochemical characteristics of oil and biodiesel that were described in accordance with ASTM D6751-02 and Mohammed et al.

## 3. Results and Discussion

### *Physicochemical Characterization of Oil/Biodiesel*

The Physicochemical characterization of cotton seed oil, waste cooking oil and its derived biodiesel was carried out. The results were obtained and presented in Table 1.

### *Evaluation of Percent Yield and Moisture Content of Samples*

CSO and WCO are the main basic ingredients used in the

transesterification process to produce biodiesel. Hexane was employed as the extracting solvent in this work to extract crude oil from cotton seeds. The percent yield of cottonseed crude oil was found to be 62.98% (Table 1) upon the extraction from the cotton seeds. Following the removal of food residues and sediments using chemical coagulation with 2% Al<sub>2</sub>SO<sub>4</sub> as a coagulating agent and gravitational sedimentation, approximately 90.24 percent of the oil was recovered. Also, there is no unreacted oil were observed on the biodiesel, which was produced from CSO and WCO. The results (see Table 1) were reveals that there is no water and sediments were found in biodiesel that were produced from CSO and WCO, even fewer amount of moisture (0.32% in CSO and 0.27% in WCO) was observed. The water and any leftover alcohol were removed using a rotator evaporator. The results of the trials showed that when constant weight was approached during the rotator evaporation, the total elimination of water and unreacted alcohol was established. According to ASTM standard limit for water and sediment is 0.05% by volume of the sample was recommended. Thus, the results shown that the biodiesel produced from, CSO and WCO were purified and refined well in this present study.

**Table 1.** Physicochemical characterizations of waste cooking oil, cotton seed oil and Biodiesel.

S.No.	Properties	Estimated values			
		CSO	Refined WCO	Biodiesel from CSO	Biodiesel from WCO
1	Yield of oil (%)	62.98	90.24	-	-
2	Moisture content (% wt.)	0.32	0.27	-	-
3	Specific gravity	0.84	0.89	0.86	0.91
4	Viscosity (mm <sup>2</sup> /sec)	36	65	4.1	5.23
5	Saponification value (mg/g)	140.25	56.1	182.33	133.237
6	Iodine value (mg I <sub>2</sub> /g)	10.659	6.091	1.01	6.048
7	Acid value (mg/KOH)	13.745	1.964	1.122	2.805
8	Free fatty acid (%)	3.95	1.88	0.226	1.128
9	Cetane Number	74.6	137.5	76	87
10	HHV (MJ/kJ)	43.52	47.04	42.122	44

### *Specific Gravity and Viscosity of the samples*

The Result shown in Table 1 Shows that the specific Gravity was reduced from 0.9125 to 0.86 on CSO and 0.9258 to 0.91 on WCO after transesterification and it with in the acceptable limit and also well agreed with ASTM Standard. The main obstacle to using vegetable and animal fats directly in diesel engines is their higher viscosity. Using a falling fluid viscometer, the viscosity of the used cooking oil was meas-

ured. The average viscosity of WCO and CSO was measured (at room temperature), according to the results to be 36mm<sup>2</sup>/sec and 65 mm<sup>2</sup>/sec respectively. The viscosity of the biodiesel was obtained from CSO and WCO were 4.1mm<sup>2</sup>/sec and 5.23 mm<sup>2</sup>/sec respectively. The results Obtained are in the range recommended by ASTM. The ASTM standard for biodiesel viscosity was reported 1.9-6.0 mm<sup>2</sup>/sec at 30 °C.

### *Determination of Saponification Value, Iodine Value, Acid*

### value & Fatty Acid Content

The saponification value of oils was observed as 182.33 mg/g (in CSO) and 133.24 mg/g (in WCO) while that of its biodiesel is 140.25 mg/g and 56.1 mg/g respectively. This suggests that the molecular weight of saturated and unsaturated fatty acids is higher in the triglycerides of CSO and WCO. The findings of this investigation are in agreement, and a similar pattern has been noted in published literature [2]. This results obtained compares favorably with the saponification value of palm oil (187-205), olive oil (185-187), and soy oil (187-193). The most crucial step in detecting adulteration is saponification.

The iodine value of CSO and WCO were estimated at 72.38 mgI<sub>2</sub> and 51.74 mgI<sub>2</sub>, this indicates that the studied oils are edible. Iodine value for edible oil is less than 100 mgI<sub>2</sub>. In general, greater the iodine value, the higher degree of unsaturation and the higher the tendency of the oil to undergo oxidative rancidity [2]. Even though its biodiesel has the iodine value of 112.4 mgI<sub>2</sub> and 120.53 mgI<sub>2</sub> respectively, which is relatively high according to Europe's EN 14214 specifications of iodine value, it indicates that the studies CSO and WCO are the good sources of raw materials for the production of biodiesel because the higher iodine value, that indicates the more number of unsaturated double bonds present in molecular structure and lower the viscosity.

ASTM standard for total acid number for pure biodiesel is reported about 0.8 mg KOH/g. The total amount of potassium hydroxide required to neutralize the free acids found in oil and biodiesel samples is known as the acid value. Lang *et al.* reported that the acid values of the ethyl esters of linseed oil, canola oil, sunflower oil and rapeseed oil were 0.884, 0.869, 0.876 and 0.873 mg KOH/g, respectively [24]. The test result of the present was found to be 0.38 mg KOH/g (in CSO), 0.50 mg KOH/g (in WCO), 0.30 mg KOH/g (in CSO biodiesel) and 0.42 mg KOH/g (in WCO biodiesel).

By managing the transesterification, cleaning, and drying procedures, this could be further enhanced. Increased acidity levels in the feedstocks suggest that the oil source is an unprocessed or inadequately refined product as a result of inadequate process control. In older engines, a higher acid number may also lead to the deterioration of rubber components and filter blockage [biodiesel-WCO]. Fatty acid contents are the major indicators of the properties of biodiesel. To determine the amount of fatty acids in the oil and biodiesel products, duplicate samples were used. The results were presented in Table 1 and it was estimated about 0.57% (in CSO), 1.9% (in WCO), its biodiesel 0.23% and 1.13% respectively. In comparison, the WCO and its biodiesel contains more FFA, among 60% fatty acid found to be monounsaturated (C18:1). Poly unsaturated fatty acids found to be 26% (C18:2 & C18:3), only approximately 8 % fatty acids were saturated. Palmitic acid and stearic acid were the major saturated fatty acids found in waste cooking oil ethyl ester are reported in literature [biodiesel-WCO].

The primary determinants of the viscosity of biodiesel are

the type and quantity of fatty acid content. Thus, implies that biodiesel derived from waste cooking of the study was observed at 5.23 which are greater than the viscosity of biodiesel derived CSO of the present study. The amount of free fatty acids in the raw materials used to produce biodiesel using an alkaline catalyst has a significant impact on the yield and quality of the final product. A high propensity to produce soap occurs when the feedstock's FFA is greater than 3% [25]. Consequently, the soap increases the viscosity of the transesterification products and makes the separation of the biodiesel from the by-products more difficult [26]. Thus, the FFA value obtained in this study was 0.23% (for CSO biodiesel) and 1.13% (for WCO biodiesel), which is within the recommended range for alkaline catalyst transesterification. The result was obtained from 0.57% and 1.88% of cottonseed oil and waste cooking oil respectively; this implies that the oil converted into methyl fatty esters. However, the level of impurities in the feedstock could be responsible for the high FFA content obtained in this study.

### Estimation of Cetane Number and Higher Heating Value of Samples

It is used to gauge how well diesel fuel ignites; the greater the cetane number, the simpler it is to start a typical (direct-injection) diesel engine. In a flammable mixture of cetane and 1 methylnaphthalene, it indicates the percentage (by volume) of cetane (chemical name hexadecane) whose ignition properties correspond to those of the diesel fuel under test. The cetane number of the biodiesel was found to be 76 (in CSO) and 87 (for WCO) which are higher than that of diesel of 48 – 65 as reflected in reported literature. High cetane number shortens the engine delay period and promotes smooth combustion. Additionally, a greater cetane number indicates more effective ignition. Biodiesel has a higher cetane number than petroleum diesel due to its higher oxygen content. With a greater cetane number, WCO biodiesel performs better in engines than CSO fuel, which lowers emissions of all pollutants except nitrogen oxides (NO<sub>x</sub>). So it is evident that the produced biodiesel possesses the positive attribute.

When a fuel burns fully and the combustion products cool back to the original temperature of the combustible mixes, the thermal energy released per unit quantity of fuel is known as the fuel's heating value. It measures the energy content in a fuel. The HHV, of the vegetable oils and their methyl esters were measured according to ASTM standard method by employing saponification value and iodine values of the oils and its methyl esters. The higher heating values (HHVs) of studied oils (40.87 MJ/kg for CSO and 43.19 MJ/kg for WCO) are good agreement with literature [HHV] and biodiesels are relatively high (shown in Table 1). The HHVs of studied biodiesel's (45.37 MJ/kg for CSO biodiesel & 48.94 MJ/kg for WCO biodiesel) is slightly lower than that of diesel (49.65 MJ/kg). Biodiesel's oxygen concentration reduces its oxidation potential and enhances combustion. A fuel's structural oxygen content increases

the fuel's oxygen homogeneity during combustion, which boosts combustion efficiency. Because of this the combustion efficiency of biodiesel is higher than that of petrodiesel and the combustion efficiency of methanol and ethanol is higher than that of diesel.

An essential characteristic that determines the energy content and, consequently, the efficiency of fuels is the HHV. It is easy to classify fuel characteristics for the combustion analysis of biodiesel blends into three categories: physical, chemical, and thermal. Physical properties include viscosity, density, cloud point, pour point, flash point, boiling range, freezing point and refractive index. There are several correlations that can be used to estimate the HHV of vegetable oils based on their physical characteristics.

## 4. Conclusions

An economical and environmentally friendly answer is provided by the manufacturing of biodiesel from cotton seed oil. The criteria used to determine approved biodiesels don't require any changes, given the variety of testing conducted for this project. The viscosity of cotton seed oil reduces substantially after transesterification and is comparable to petrodiesel and the physical and chemical properties of biodiesel produced conform to EN/ASTM standards. Analysis was done on how various parameters, including temperature, catalyst concentration, reaction time, and reactant ratio, affected the amount of biodiesel produced. We may draw the conclusion that while utilizing water as a cleaning agent improves product purity, it has no effect on reaction productivity. The molar ratio of alcohol to oil and the catalyst concentration are the two special factors that influence the generation of biodiesel. According with the above, the best conditions of operation are: Molar ratio of alcohol to oil 6:1, Catalyst concentration: 1%w/w, Reaction temperature: 60 °C, Washing agent: water at 40 °C. Compared to diesel, biodiesel has a lower elemental composition. As a result, biodiesel produces fewer pollutants than diesel. Due to its low levels of free fatty acids, cotton seed oil can be produced economically on a wide scale without the need for a two-stage alkaline transesterification process to produce biodiesel. The properties are similar to those of diesel with the additional benefits of a high cetane number of 76 hence the biodiesel can be used as alternative fuel for diesel engines.

## Abbreviations

ASTM	American Society for Testing Materials
CS	Cotton Seed
EN	European Norm
FFA	Free Fatty Acid
HHV	High Heat Value
RPM	Rotation Per Minute
SNNPR	South Nation Nationality and Peoples Region

WCO Waste Cooking Oil

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## Conflict of Interest

The authors declare no conflicts of interest.

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