

Production and Property Analysis of Biodiesel from Cotton Seed Oil Using Natural Catalyst

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Abstract

Nowadays as we are seeing that consumption of fossil fuels is increasing day by day and these fossils fuels are present in limited quantity on earth's surface. India's energy use is the fourth highest in the world. Biodiesel and bioethanol appear to be promising future energy sources. Biodiesel, the most promising alternative diesel fuel, has received considerable attention in recent years due to its following merits: biodegradable, renewable, non-toxic, less emission of gaseous and particulate pollutants with higher cetane number than normal diesel. In this work biodiesel was prepared from cottonseed oil by transesterification with methanol, using calcium oxide as catalysts. Calcium oxide catalysts were prepared from natural calcium sources such as mussel shell and the catalytic activities were evaluated in the transesterification of cotton seed oil. The catalysts were characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning electron microscopy (SEM) methods. Physical and chemical properties of cotton seed methyl ester are compared to that of petroleum diesel.

INTRODUCTION

The depletion of world petroleum reserves due to rapidly growing energy demands coupled with environmental concerns has promoted the efforts to explore some alternative sources of petroleum based fuels. Fuels are great importance because they can be burned to produce significant amounts of energy. The major percentages of energy used in the world today are being generated from fossil fuel sources. Fossil fuel contributes 80% of the world's energy needs

Alternative fuel such as vegetable oils can be used as sustainable fuels. There are several advantages of using vegetable oils as fuels such as higher energy content than other energy crop like alcohols. Vegetable oil is one of the renewable fuels. Vegetables oils have become more attractive recently because of its environmental benefits and the fact that it is made from renewable resources. The vegetable oils have 90% energy content of diesel. The current prices of vegetable oils in world are nearly competitive with petroleum fuel prices but these are not economically feasible yet and need further R&D work for development work. Vegetables oils can be successfully used in CI engine through engine and fuel modification. Engine modification includes dual fueling

injection modification system, heated fuel lines and blending of vegetable oil with diesel.

A. BIODIESEL

Biodiesel is a biodegradable and non-toxic fuel produced from vegetable oil and animal fats which are renewable. It can be used in C.I engine without modification. The biodiesel combustion by products are better not only for inhabitants but also for earth's environment. The emissions of unburned hydrocarbons, carbon monoxide and particular matter of biodiesel combustion are much lesser than conventional diesel fuel. 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. Biodiesel tends to freeze or crystallize in cold weather conditions and may be unsuitable to be use in engine. In order to criticize biodiesel as an alternative fuel, people used this point. But there are many techniques by which one can use biodiesel even in the cold weather conditions. Biodiesel is safer in handling and in storing as a fuel, because its flash point is more than petroleum based fuel. The cost of various feedstock of biodiesel is different as per availability and as per production technology. As the demand will increase the cost of biodiesel will also decrease. One of the major reasons to have favorable conditions to use biodiesel as alternative fuel is the cost of crude oil which is very high and varying on daily basis. This leads to a growing in the field of biodiesel in developed and in developing countries. Because of all such type of problems vegetable oils must be modified chemically in to the suitable fuel to an engine.

B. BIODIESEL FEEDSTOCKS

Cotton & Cotton seed

Cotton is one of the most important commercial crops of India and is the single largest natural source of fibre. A moderate temperature of 25-35 degree Celsius is best suited for cotton cultivation in India. It is processed in huge quantity through modern machines based on the quality requirement. The reason for such a huge production is the climate that is most favorable in northern part of the country.



Figure 1. Cotton seed

C. BIODIESEL PRODUCTION METHODS

Transformation of vegetable oils into biodiesel can be realized using four technologies:

- Dilution/blending.
- Micro-emulsion.
- Heating/pyrolysis.
- Transesterification.

Among all these techniques, the transesterification is an extensive, convenient and the most promising method for the reduction of viscosity, density and other properties of the straight vegetable oils. It was conducted as early as 1853 by scientists E. Duffy and J. Patrick.

CATALYST PREPARATION AND CHARACTERIZATION

Calcium oxide is one of the most promising heterogeneous alkali catalysts since it is cheap, abundantly available in nature (as limestone) and some of the sources of this compound are renewable (waste material consisting of CaCO_3).

A. CATALYST PREPARATION

The mussel shells were cleaned and washed thoroughly with warm water several times. The washed mussel shells were calcined at 1000°C with heating rate of $10^\circ\text{C}/\text{min}$ for 1 h. The solid powder was crushed and sieved with $63\ \mu\text{m}$. This resulted solid is as known as non-impregnated catalyst. The impregnated catalyst was prepared by the impregnation process using potassium hydroxide. A typical calcium oxide was suspended in ammonia solution (5 wt. %), and 50 mL aqueous potassium hydroxide was added. The solution was heated at 80°C for 2 h. The precipitate was collected, washed and calcined at 400°C for 12h.

B. CATALYST CHARACTERIZATION

The qualitative analysis of the sample was done using FTIR spectroscopy technique. Thermo Scientific (Model: Nicolet IS5) combined with Omnic software was employed to analyze the sample.

MATERIAL AND METHODS

A. EXPERIMENTAL SETUP

The experimental setup consists of a Rota mantle, condenser tube, round bottom flask and a pump for supply coolant (water). The Rota mantle along with the condenser is shown in fig 2. The Rota mantle has a heating mantle which is used to maintain a coolant temperature. A heat control knob is provided to control the temperature. A round bottom flask is placed on the Rota mantle in which the reaction takes place. An electromagnetic stirrer is used to mix the reactants uniformly. A speed control knob is provided the speed of the stirrer.

B. EXTRACTION OF COTTON SEED OIL

Cottonseed oil processing mainly including the flowing production process, they are seed cleaning, cracking, flaking, cooking, expelling and refining.

C. FILTRATION OF COTTON SEED OIL

Getting rid of organic impurity, inorganic impurity, oiliness impurity. In this section we use Magnetic Separator, Vibrating Sieve, destoner and decorticator. Through cleaning the impurity, you can reduce the loss of oil & improve the oil yield and the environment of production plant. The material has been produced after pretreatment contains the impurities $\leq 0.1\%$.

D. DETERMINATION OF ACID VALUE

For 1000 ml distilled water 56.11 g/mol of KOH is added at 0.1 normality. The solution is made by adding 5.611 g/mol of KOH to 1000 ml distilled water at 0.1 normality. 100ml of the made up solution is taken in the burette. 1 gram of cotton seed oil and 1 or 2 drops of phenolphthalein indicator are taken in a conical flask and mixed. 1 gram of oil was titrated against standard potassium hydroxide solution using phenolphthalein indicator. The acid value of the oil was determined by knowing the equivalent weight of KOH. Determination of acid value is shown in fig 3.

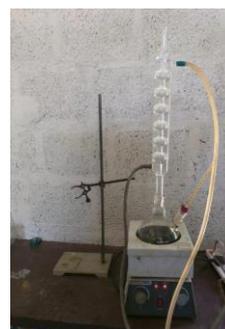


Figure 2. Rota mantle with condenser tube



Figure 3. Determination of Acid value calculation

$$\text{Acid number} = \frac{\text{Titration value} \times \text{Molecular wt. of KOH} \times 0.1}{\text{Normality of KOH} \times \text{Weight of oil}}$$

The acid value of raw oil is determined by dividing the acid number by 2 as shown in eqn. from which the free fatty acid (FFA) contents of oil is also found out. The FFA contents are determined using the formula.

$$\text{Free Fatty Acid} = \text{Acid value}/2$$

E. ACID ESTERIFICATION

Oil is taken in around bottom flask and it is heated to 650C. Methanol and sulphuric acid are mixed separately and stirred. Then the mixture is added with the oil in the setup. Stirrer is switched on and the reaction takes place for 90 minutes. After 90 minutes, the product is removed and allowed to settle in a separating flask for 8 hours. The methyl ester is obtained at the lower portion and the top portion is to be removed. The determination of acid esterification is shown in fig 4.

The acid value of the esters is determined. The esterification process is carried out for various concentrations of H₂SO₄ and oil to methanol ratio. The acid value and free fatty acid contents are determined in each case. Fig shows the settling of the products of esterification, where the methyl ester is obtained at the lower portion.

F. BASE TRANSESTERIFICATION

The transesterification process is catalyzed by alkaline metal alkoxides and hydroxides. Alkaline metal alkoxides are the most active base catalysts since they give very high yields (>98%) in short reaction time (30 min) even if they are applied at low molar concentrations. Calcium oxide is the most widely used biodiesel catalyst with over 60% of industrial plants using this catalyst. The fig 5 shows the base transesterification.



Figure 4. Acid Esterification

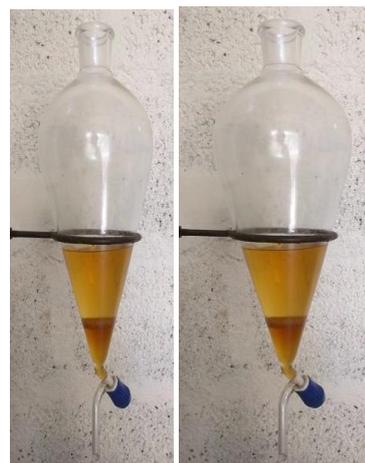


Figure 5. Base transesterification

The methyl ester produced by acid esterification is transferred into a biodiesel reactor and then the catalyst/alcohol mixture is pumped into the oil. The final mixture is stirred vigorously for 45 minutes at 550C in ambient pressure. A successful base esterification reaction produces two liquid phases ester and crude glycerol as shown in fig. The yield of biodiesel is determined. The base catalyst esterification is repeated for varying concentration of the base catalyst and methanol. Yield of biodiesel is determined in each case.

G. BIODIESEL PURIFICATION PROCESS

The reaction mixture after base esterification is allowed to settle in the reaction vessel in order to allow the initial separation of biodiesel and glycerol or the mixture is pumped into a settling vessel. The glycerol phase is much denser than the biodiesel phase and settles at the bottom of the reaction vessel, allowing it to be separated from the biodiesel phase. Understanding the limits of biodiesel purification methods and selecting appropriate combinations of feedstock and purification steps, will lead to a consistent quality of fuel at a cost both the producer and customer can afford.

H. RECOVERY OF GLYCEROL

The entire mixture in the settling tank then settles and glycerol is left on the bottom and a methyl ester (biodiesel) is left on top. Phase separation is completed in 2 hours after stirring. Complete settling can take as long as 18 hours. After settling is complete, water is added at the rate of 5% by the volume of the oil then stirred for 5 minutes and the glycerol is allowed to settle again. After settling is complete the glycerol is drained and the ester layer remains. The recovery of high-quality glycerol as a biodiesel by product is a primary action to be considered lowering the cost of biodiesel. By neutralizing the free fatty acids, removing the glycerol and creating an alcohol ester, transesterification occurs. This is accomplished by mixing methanol with sodium hydroxide to make sodium methoxide. This dangerous liquid is then mixed with vegetable oil.

Washing the methyl ester is a two step process that is carried out with extreme care. This procedure is continued until the methyl ester layer becomes clear. After settling, the aqueous solution is drained and water alone is added at 28% by volume of oil for the final washing. The resulting biodiesel fuel, when used directly in a diesel engine, will burn upto 75% cleaner than D2 fuel.

I. Purification of Crude Biodiesel

Dry washing is an interesting method, on small scale, as no water is required. Adsorbents such as Magnesol (a commercial adsorbent composed of amorphous magnesium silicate) or ion exchange resins are used to eliminate water and other contaminants from biodiesel. One of the main limitations of such adsorbents is their adsorbing capacity. It is essential to have eliminated most of the residual alcohol during the earlier purification stages to avoid saturating the adsorbents.

The contaminants can be removed by using a hollow fiber membrane extraction, such as polysulfone. In this method, a hollow fiber membrane(1m long, 1mm diameter) filled with distilled water is immersed into the reactor. The crude biodiesel is pumped into the hollow fiber membrane. Following this step, biodiesel is passed over heated Na₂SO₄ and then filtered to remove any remaining water. This approach effectively avoids emulsification during the washing step and decreases the loss during the refining process. The purity of the biodiesel obtained about 90% and the other properties conform to the ASTM standard. It is a very promising method for purifying biodiesel. The pure biodiesel obtained at the end of purification process.

RESULT AND DISCUSSION

The various property test results of both raw cotton seed and the biodiesel are compared with the ASTM and EN standards and the discussions are made.

A. ACID VALUE CALCULATION

Acid value = 45.36mg KOH/g

The measured acid value of cotton seed oil is higher than the standard acid value (2mg KOH/g) of the oil. The free fatty acid content in the cotton seed oil is higher. So the biodiesel is produced by acid esterification and base transesterification.

B. OIL ANALYSIS

Table 1. Oil analysis of the cotton seed oil

Content	Result %	Method
Free fatty acid	22.39	AOCS Cd 3d-63
Triglycerides	75.545	ASTM D6584
Sediment	0.833	AOCS Ca 3d-02
Moisture	0.23	AOCS Ca 2e-84

The table represents the oil analysis of the cotton seed oil. The cotton seed oil has 22.39% of fatty acid which was measured by AOCS Cd 3d-63. It has 75.545 % of the triglycerides which was measured by ASTM D6584. The rate of the sediments and moisture in the cotton seed oil is 0.833% and 0.23% which were measured by AOCS Ca 3d-02 and AOCS Ca 2e-84.

Kinematic viscosity

Viscosity of Cotton seed oil was founded out by using Redwood viscometer which is 38.65mm/s at 40°C. According to the EN ISO 3104 biodiesel standard the viscosity should be 3.5 – 5.0mm²/s.

Density

The density of cotton seed oil is found to be 920kg/m³. According to the EN ISO 3104 standard biodiesel standard the density should be 850 – 900mm²/s. The density of Cotton seed oil will further reduced when it undergoes heating transesterification process.

Flash point and fire point

Flash and Fire point of cotton seed oil was founded by using Cleave land flash and fire point Apparatus. Flashpoint of cotton seed oil is 225°C and the fire point is 310°C. According to the EN ISO 3679 biodiesel standards the flash point should be more than 120°C. Therefore cotton seed oil met the required flash point and fire point.

C. ACID ESTERIFICATION

The free fatty acid of cotton seed oil is 22.39%. In acid transesterification, the free fatty acids are converted to the esters. 100 ml of refined cotton seed oil is poured into the two necks round bottle and heated upto 700C. The 33% of the methanol and 0.5 % of the H₂SO₄ (Sulfuric acid) mixture is poured into the bottle. Heating and stirring should continue about 45 min at atmospheric pressure. Finally the mixture is poured into the separating funnel for separating the excess alcohol, impurities and sulfuric acid. The excess alcohol, sulfuric acid and impurities move to the bottom layer of the separating funnel and it is discarded. The top layer is separated for further processing. The above procedure is repeated three times for reducing the acid value of cotton seed oil. This process reduces the acid value of refined cotton seed oil to less than 2 % of FFA.

D. ALKALI TRANSESTERIFICATION

The triglycerides value of cotton seed oil is 75.545%. During the alkali transesterification process, these triglycerides are converted to biodiesel. After acid pretreatment the esterified oil is taken in the two necks round bottle and heated upto 700C. 5% of CaO is dissolved in 33% of methanol. The dissolved solution is poured into flask. The mixture is heated and stirred for 90 min and completion of reaction, the mixture into the separating funnel over 12 hours. The glycerol and impurities are settled in bottom layer of the separating funnel and it is discarded. The impure biodiesel remain in upper layer. It contains some trace of catalyst, glycerol and methanol. The washing process can be done by the 3/4th of hot distilled water added with methyl ester and gently stirred. The upper layer is pure biodiesel and lower layer is drawn off.

Catalyst characterization

The SEM and FTIR test result of the Calcium Oxide catalyst are discussed below.

Fourier Transforms Infrared Spectrometer(FTIR)

FTIR spectroscopy, as an effective tool for a semi-quantitative estimation of structural information in complex solids, is used to investigate the functional groups in the catalyst. FTIR spectra of all particle sizes were recorded and studied in the wave number range 500-4000 cm⁻¹. Prominent absorption peaks were obtained at wave number 3934.78, 3695.61, 3525.88, 3444.87, 2983.88, 2875.86, 2515.18, 1795.73, 875.68, 711.73, and 428.20cm⁻¹. The peak at 3934.78, 3695.61, 3525.88, and 3444.87cm⁻¹ were due to amide N-H medium C=O strong. The bands at 3934.78, 3695.61, 3525.88 and 3444.87cm⁻¹ were assigned to the amines and amides present in mussel shell.

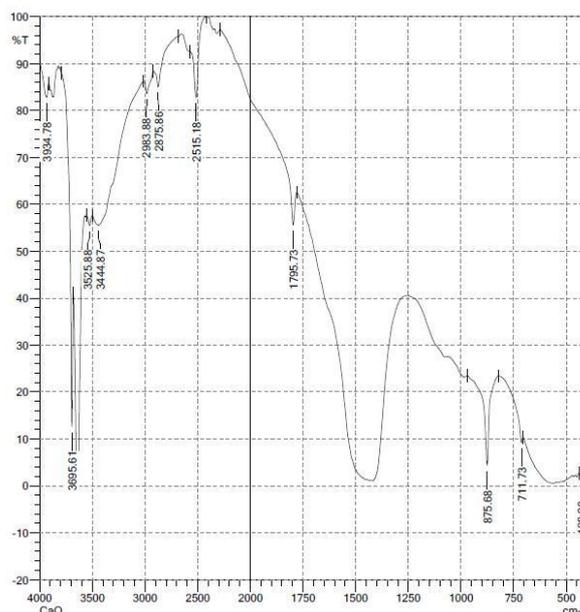


Figure 6. Fourier Transforms Infrared Spectrometer (FTIR)

The peak at 2983.88, 2875.86 cm⁻¹ was due to C-H (sp³ carbon) strong, broad and multi-banded. The peak at 2515.18cm⁻¹ is due to carboxyl acid C=O&O-

H stretch strong. The peak at 1795.73cm⁻¹ is due to acid chlorides C=O stretch very strong. As per report, absorption peaks in the range 780 - 900 cm⁻¹ are due to antisymmetric stretching of O - Al - O and peaks in the range 550 - 650 cm⁻¹ are due to antisymmetric bending. It's evident that Ca-O group absorptions were strongly attributed over wave numbers <600cm⁻¹. The peak value at 875.68, 711.73 and 428.20 cm⁻¹ indicates plane bending vibration of carbonate.

Scanning Electron Microscope (SEM)

Scanning electron microscope is a very useful tool for studying morphology of Nano powders. The surface morphology of the CaCO₃ Nano powder synthesized was examined by SEM (scanning electron microscope) operating at 20kV accelerating voltage. The CaCO₃Nano powder is a non-conducting powder therefore the SEM images were taken at low vacuum mode to obtain sharp images. The images are shown in Figure. Calcite and aragonite possess different crystal growth patterns and crystal structure. Calcite is hexagonal and aragonite is orthorhombic. Cube-like crystals of calcite are stable as compared to Rod-like orthorhombic crystals of aragonite. These figures show that, the particles are separate; hence the CaO particles seem to be dispersing and form tininess with a smooth particles surface. The separated particles and the bulk morphology of the CaO may be the reason for the separation difficulties occurred when processing CaO in biodiesel production.

E. GC-MS ANALYSIS

Table 2. GCMS Analysis

RT	Name of Free Fatty Acid	Molecular Formula	Molecular weight	Relative %	
4.05	C16:0	Palmitic acid	C17H34O2	270	0.65
4.35	C14:0	Myristic acid	C14H28O2	228	0.29
5.12	C16:1	Palmitoleic acid	C16H30O2	254	0.25
7.42	C15:0	Pentadecylic acid	C17H34O2	270	11.33
9.44	C17:0	Margaric acid	C19H38O2	298	0.42
12.32	C18:2	Linoleic acid	C19H34O2	294	42.70
13.52	C18:0	Stearic acid	C20H38O2	310	2.30
16.03	C18:1	Vaccenic acid	C19H36O2	296	39.03
19.33	C21:0	Heneicosylic acid	C21H42O2	338	1.08
24.26	C18:2	Linoleic acid	C19H34O2	294	0.21
29.34	C20:0	Arachidic acid	C20H38O2	310	0.61
34.22	C20:1	Paullinic acid	C21H40O2	324	1.13

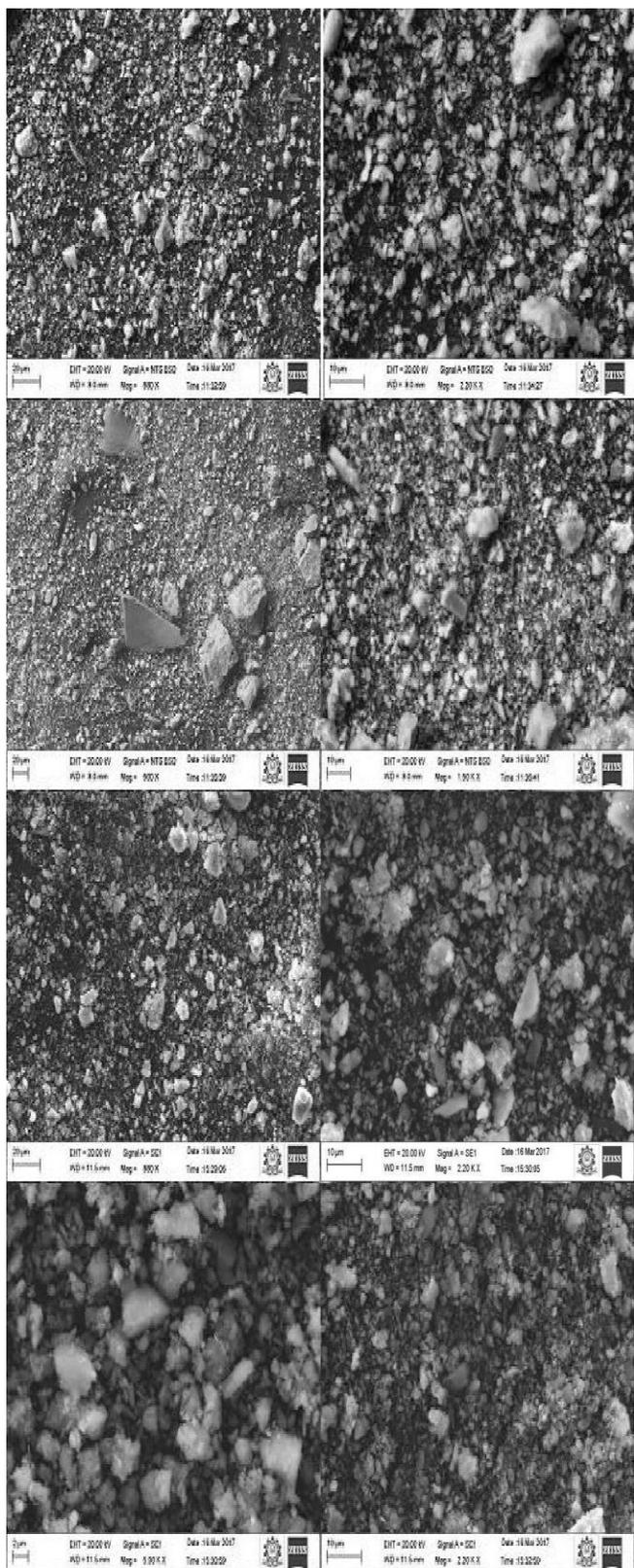


Figure 7. Scanning Electron Microscope image

The fatty acid compositions of the oils seem to have an important role in the performance of the biodiesel in diesel Acids engine. The unsaturated fatty chains, especially the polyunsaturated species (linoleic acid), are susceptible to oxidation, i.e. reaction with the oxygen in ambient air. Oxidative stability and combustion properties of biodiesel improve with increasing saturation of the fatty acid chains. Cold flow property increases with the increasing of the unsaturation.

The Cetane number decreases with increasing number of carbon atoms and increase in unsaturation. Saturated long-chain esters such as methyl palmitate and methyl stearate have high Cetane number. The kinematic viscosity increases with the number of CH₂ moieties in the fatty ester chain and decreases with an increasing unsaturation. The bulk saturated fatty acid are responsible for high viscosity of the diesel.

The higher level of unsaturated fatty acids reduces the fuel quality. The cotton seed oil has 57.818% of saturated acid, 25.216% of Monosaturated acid and 16.964% of polysaturated acid. Its shows that the oil has the saturated fatty acids higher than the both mono and poly saturated fatty acids. The presence of higher saturated fatty acid leads to high viscosity, high Cetane number and better biodiesel stability. So the biodiesel from the cotton seed oil has good oxidative stability, high viscosity, high Cetane number and good stability.

Oxidative stability

Oxidative stability of biodiesel is extremely important property, especially in places with warm climate, as these properties provides an estimate of the storage time of biodiesel in normal conditions. Esters with large amounts of unsaturated fatty acids are much more susceptible to oxidation than those that are rich in saturated fatty acids. The minimum oxidation stability time of EN 14112 and ASTM 6751 at 1100C are 3 hours and 4 hours. Here the oxidation stability time of cotton seed biodiesel at 1100C (5.2760 hours) exceeds the minimum limit of the EN 14112 and ASTM 6751 standards. So this biodiesel is suitable for the fuel applications.

Iodine value

The iodine value provides the information about the unsaturation degree of the oil which directly affects its stability to oxidation. The reason for auto-oxidation occurrence is the presence of double bonds in the chain of the fatty compounds. Hence, the limitation of unsaturated fatty acids in biodiesel is necessary. Iodine value is limited to 120 g I/100g in the American

Society for Testing and Materials (ASTM 6751) and limited to 130g I/100g in the European biodiesel standards (EN 14111). The biodiesel of cotton seed oil (119 g I/100g) has less iodine value than the both ASTM 6751 and EN 14111 biodiesel specifications, thus they have moderate degree of unsaturation and they are suitable for fuel application.

F. BIODIESEL PROPERTY

Table 3. Properties of Biodiesel

Property	Cotton seed biodiesel	Petro-diesel	EN		ASTM 6751		Units
			Protocol	Value	Protocol	Value	
Oxidative stability, 1100C	5.276	-	EN 14112	Min 4	ASTM D 6751	Min 3	hours
Iodine number	119	-	EN 14111	Max 130	-	Max 120	g I/100g
Cetane number	67.21	49	EN ISO 5165	Min 51	D-613	Min 47	-
Carbon residue	0.782	0.275	EN ISO 10370	Max 0.30	ASTM D 6751	0.05	%
Flash point	162	70	EN ISO 2719	Min 55	D-93	Min 130	°C
Density @ 300C	895	825	EN ISO 3675	Min 860	D-93	870-900	Kg/m ³
Kinematic viscosity @400	5.8	2.25-3.5	EN ISO 3104	2-5	D-93	1.9-6.0	mm ² /s

CONCLUSION

This study revealed that biodiesel could be produced successfully from the cottonseed oil by acid esterification and alkali-catalyzed transesterification. A low cost calcium oxide

catalyst developed from mussel shells. The production of biodiesel from cotton seed oil offers a double-facet solution: economic, environmental. The viscosity of cotton seed oil reduces substantially after transesterification and is comparable to petro-diesel. The physical, chemical and fuel properties of biodiesel were conformed to EN/ASTM standards. SEM helped to determining the surface morphology; FTIR detected the existing functional groups. The catalyst showed excellent performance in biodiesel production. Important fuel properties of COME such as cetane number, kinematic viscosity, and acid value, free and total glycerin were compared well with ASTM and EN specifications. The production of COME from native cottonseed oil may simultaneously reduce dependence on imported fossil fuels and help to alleviate the food versus fuel dilemma that plagues rapeseed, soybean, palm, and other oil seed crops that are also traditional food sources. Biodiesel produced from cotton seed oil possess a great potential for being source of alternate fuel.

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