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Research Article

Processing and Characterization of Cotton Seed Methyl Ester

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Abstract

Production of cotton seed methyl ester (biodiesel) from non-edible vegetable oils for diesel substitute is particularly important because of the decreasing trend of economical oil reserves, environmental problems caused due to fossil fuel use and the high price of petroleum products in the international market. Present work reports an optimized protocol for the production of methyl ester through alkaline catalyzed transesterification of cotton seed oil. Three principal variables, molar ratio of methanol to oil, amount of catalyst, and reaction temperature affecting the yield of alkaline catalyzed production of methyl ester from cotton seed oil were investigated. The reaction variables used were methanol/oil molar ratio (4:1–8:1), catalyst concentration (0.5–2%), temperature (50–70°C), and catalyst type. The rate of transesterification in a batch reactor increased with temperature up to 60°C. The methyl ester with best yield and quality was produced with cotton seed methyl ester at 6:1 mole ratio and 1 wt% of catalyst (N_aOH) the yield was 93 %. It was noted that greater or lower the concentration of N_aOH or methanol than the optimal values, the reaction either fully occur or lead to soap formation. Physical and chemical properties of cotton seed methyl ester are compared to that of petroleum diesel. The produced methyl ester was found to exhibit fuel properties within the limits prescribed by the American Standards for Testing Material (ASTM) and European EN standards.

Keywords: Cotton seed oil; Methyl ester; Physicochemical properties; Renewable sources; Transesterification

Introduction

Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as methanol [1,2,3]. The reaction requires a catalyst, usually a strong base, such as sodium or potassium hydroxide, and produces new chemical compounds called methyl esters. It is these esters that have come to be known as biodiesel. Because its primary feedstock is a vegetable oil or animal fat, biodiesel is generally considered to be renewable [4,5]. Since the carbon in the oil or fat originated mostly from carbon dioxide in the air, biodiesel is considered to contribute much less to global warming than fossil fuels [4]. Diesel engines operated on biodiesel have lower emissions of carbon monoxide, unburned hydrocarbons, particulate matter, and air toxics than when operated on petroleum based diesel fuel [6,7]. The use of edible vegetable oils and animal fats for biodiesel production has recently been of great concern because they compete with food materials. As the demand for vegetable oils for food has increased tremendously in recent years, it is impossible to justify the use of these oils for fuel use purposes such as biodiesel production. Moreover, these oils could be more expensive to use as fuel. Hence, the contribution of non-edible oils such as cottonseed and coconut will be significant as a non-edible plant oil source for biodiesel production [8]. In recent years, there exist active researches on biodiesel production from cottonseed oil of which the conversion between 72% and 94% was obtained by enzyme catalyzed transesterification when the refined cottonseed oil reacted with short-chain primary and secondary alcohols [9,10,11]. The application of solid acid catalysts on cottonseed oil transesterification. The results showed that the yield of methyl ester was above 90% after 8 hours of reaction [12]. In Contrast, transesterifying cottonseed oil by microwave irradiation could produce a biodiesel yield in the range of 89.5-92.7% [13]. No matter what kind of catalysts or approaches were applied, all those studies aimed to produce high yield of biodiesel by optimized reaction conditions based on optimized parameters in terms of alcohol/oil molar ratio, catalyst concentration, reaction temperature, and time. However, nearly in all studied cases, there existed complex interactions among the variables that remarkably affected the biodiesel yield [14,15,16].

The most common way of producing biodiesel is the transesterification of vegetable oils. The methyl ester produced by transesterification of vegetable oil has a high cetane number, low viscosity and improved heating value compared to those of pure vegetable oil which results in shorter ignition delay and longer combustion duration and hence low particulate emissions [17]. Its use results in the minimization of carbon deposits on injector nozzles [18]. The main Objectives of the work are to study the processing parameters which effecting the production yield of biodiesel from cotton seed oil and to evaluate the physical and chemical characteristics of biodiesel produced.

Materials and Methodology

In the present investigation experimental work was carried out in two levels. In the first level process development studies using cotton seed vegetable oil were undertaken. In the second level biodiesel samples obtained in the experimental work were evaluated.

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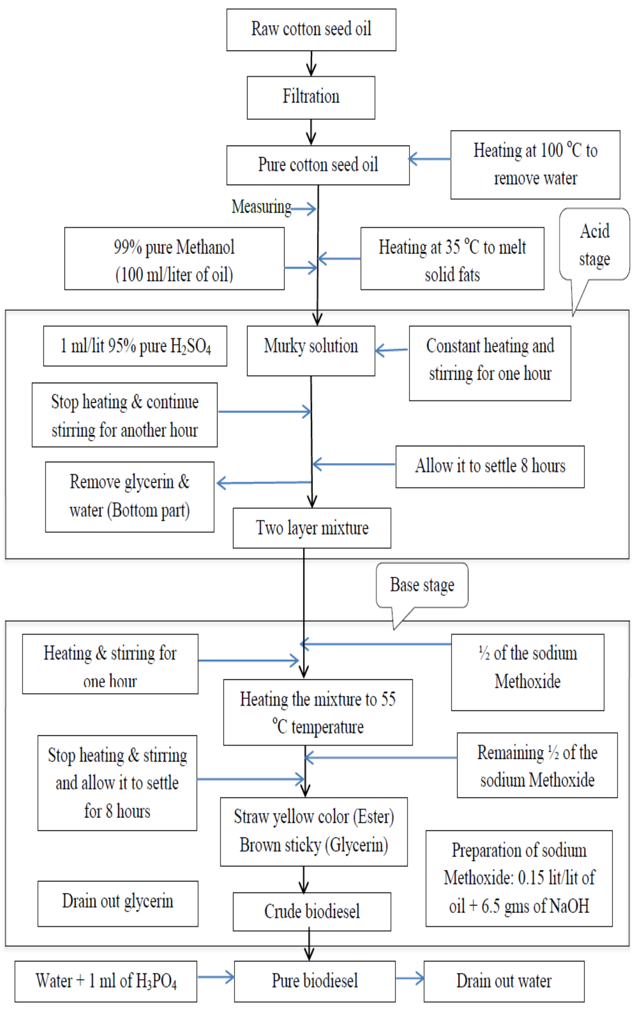

Figure 1: Flowchart for Biodiesel production.

The process development studies are divided into three steps:

1. Preconditioning of oil
2. Transesterification reaction
3. Purification of products

The procedure of biodiesel from cotton seed oil is from flowchart shown in (Figure 1).

Preconditioning of oil

Preconditioning of oil involved the removal of the moisture and neutralization of free fatty acids (FFA). Usually the present feed (cotton seed oil) consist less than 5% of FFA but they need to be neutralized as they result in the formation of soap. For determination of FFA present in the oil taken, we titrated the oil sample with 0.1N KOH solution with Phenolphthalein as indicator. Volume in ml of 0.1N KOH required to neutralize 1gram of oil is called acid value, and with this the amount of KOH needed to neutralize the FFA's in the oil. Thus by adding calculated amount of KOH, FFA's are removed in the form of soap. Removal of moisture involved heating the oil sample up to 105-120°C and maintaining it there for few minutes so that all moisture gets evaporated. If required vacuum pump arrangement is used to remove the moisture present.

Trans-esterification reaction

Transesterification reaction takes place in two stages.

First stage (Acid catalyzed stage): The free fatty acids can be reduced to esters by two processes viz. hydrolysis, methanol with acid catalyst. The two reactions are depicted in the (Figure 1). The treated oil is taken in liters and heated to 35°C to melt the solid fats presented in the oil. Methanol of 99% pure is added (0.1 liters/liter of oil) to the heated oil. It is stirred for five to ten minutes (methanol is a polar compound; oil is strongly non-polar; hence a suspension will form). One milliliter of 95% pure sulfuric acid (H_2SO_4) is added for each liter of oil using a graduated eye dropper [13]. The compound is stirred for one hour maintaining the temperature at 35°C. Heating is stopped and the mixture is stirred for another hour. The mixture is allowed to settle for eight hours in a decanter to remove glycerin and chemical water [19].

Second stage (Base catalyzed stage): For each liter of oil/fat 0.12 liter of methanol (12% by volume) is measured to which it is added 6.5 grams/ liter of lye (N_aOH). The mixture is stirred thoroughly until it forms sodium methoxide. Half of the prepared sodium methoxide is poured into the unheated mixture and the mixture is stirred for five minutes. This will neutralize the sulfuric acid. The mixture is heated to 55°C and the whole reaction is maintained. Remaining sodium methoxide is added to the heated mixture and stirred at low speed of not more than 500 to 600 rpm. After one hour the mixture is poured into a decanter and allowed it to settle for 8 hours. As glycerin is heavier than the methyl ester (biodiesel), it will settle at the bottom. The glycerin is separated from the methyl ester (biodiesel) [20].

Purification of products

Bubble wash method is used; one milliliter of phosphoric acid (H_3PO_4) is added to the washing water first. One third water by volume to the oil is being used and the oil is bubble washed for twenty hours. The mixture is allowed to settle in a decanter for one hour and the water is drained off latter. The biodiesel is heated to 100°C to dispense with the traces of water and preserved. Samples obtained under optimum conditions, are characterized to know the physico-chemical properties of the cotton seed biodiesel, the characterization of biodiesel is also explained [21,22,16].

Results and Discussions

Influence of processing parameters on cotton seed methyl ester (biodiesel) yield

Biodiesel production from cottonseed oil was carried out for different parameters like catalyst type (N_aOH), catalyst concentration (0.5%, 0.75%, 1%, 1.25%, 1.5%,1.75 and 2%), Reaction time (45, 60, 75, 90, 105,120 min), Reaction temperature (50, 55, 60, 65 and 70°C) and Molar ratio alcohol to oil ratio (4:1, 5:1, 6:1, 7:1, 8:1).

Influence of catalyst concentration on biodiesel yield

It shows that the mean yield increased when the catalyst amount was increased from 0.5 to 1.0 wt%. On the contrary, the mean yield decreased significantly when the catalyst amount exceeded 1.0 wt%. Two reasons may be offered to explain why catalyst amount 0.5 and 1.5 wt% resulted in lower mean yield. First, cotton seed oil contains a trace of FFA. During the transesterification reaction, some of the

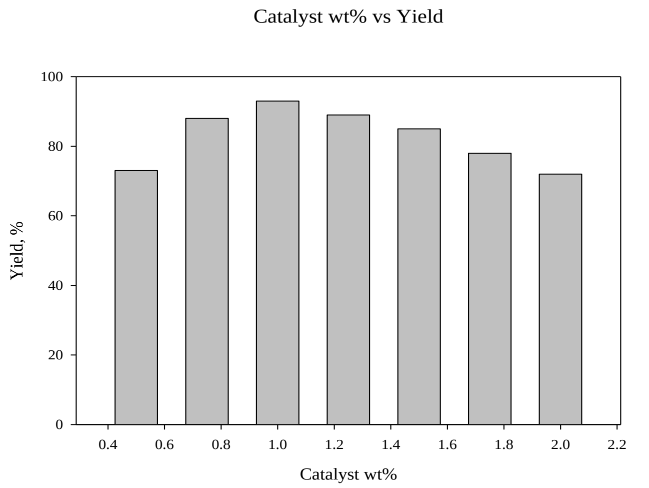

Figure 2: Biodiesel yield with catalyst concentration.

catalysts were neutralized by FFA. Therefore, increasing the amount of catalyst used can improve the yield. This might explain why the mean yield was only 73 % when 0.5 wt% of the catalyst was used and why the mean yield increased with when catalyst amount was increased from 0.5 to 1.0 wt%. Second, too much catalyst used might initiate a saponification reaction. This might explain why the use of 1.5 wt% of the catalyst resulted in a lower mean yield than 1.0 wt%. In short, there is an optimal amount of catalyst to be used at 1.0 wt% catalyst gives 93 % yield. This is the optimal level of catalyst amount. The result was consistent with earlier findings that catalyst amount is the significant factor on the yield of non-edible oil, and the optimal reaction levels should be around 1.0 wt%. Conversion graph is as shown in (Figure 2).

Influence of temperature on biodiesel yield

The primary advantage of higher temperatures influences to shorter reaction time. However, higher reaction temperatures causes methanol to vaporize resulting in decreased yield. In this process, the temperature ranges used 50, 55, 60, 65, and 70°C and in order to optimize biodiesel yield with using the constant parameters are: alcohols to oil molar ratio of 6:1 with NaOH concentration of 1 wt% for cottonseed oil at 90 min reaction time. Describes biodiesel yield with respect to temperature ranges of 50, 55, 60, 65, 70°C for cottonseed oil. 50°C shows, biodiesel yield were 82%for cottonseed oil. Again increases the temperature to 55°C and biodiesel yield also increases. Further the temperature increases to 60°C, the biodiesel yield were 93% for cottonseed oil. When the temperature decreases, the rate of reaction also decreases. Therefore, the equilibrium concentration was strongly conditioned by the temperature and favored for the same; i.e., the equilibrium concentration increased as the temperature increased. Therefore the optimum temperature is 60°C for cottonseed oil biodiesel process. Ester Conversion graph shown in (Figure 3).

Influence of reaction time on biodiesel yield

According to many researchers, the biodiesel yields are directly proportional to the reaction times used. This experiment was conducted from the reaction time of 45, 60, 75, 90,105 and 120 min with the constant parameters: 60°C of reaction temperature for 1 wt

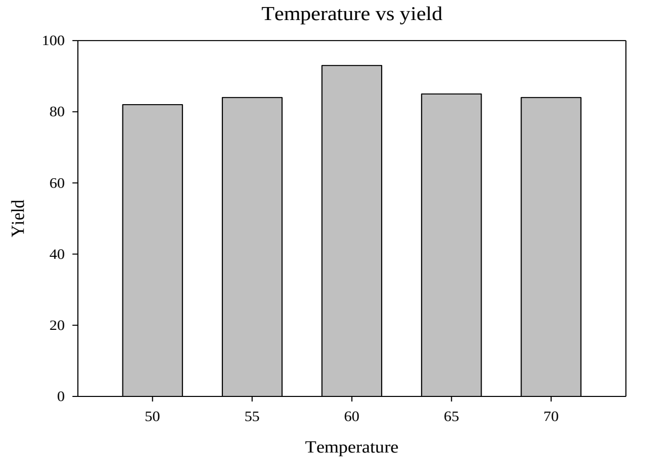

Figure 3: Biodiesel yield with temperature.

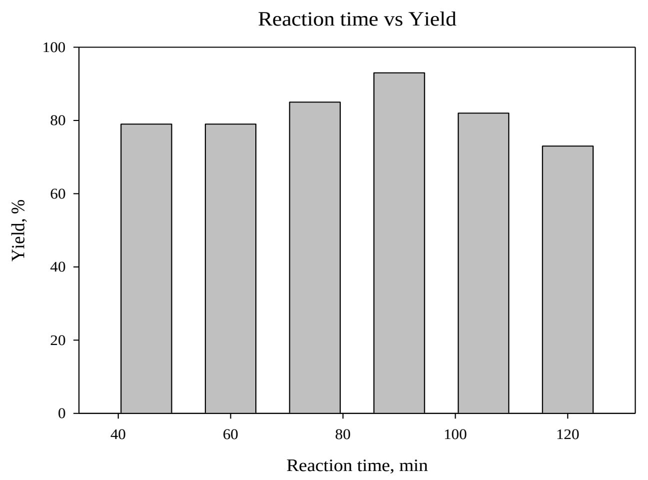

Figure 4: Biodiesel yield with reaction time.

% of N_aOH catalyst used in 6:1 molar ratio of cottonseed oil show that effect longer mixing gives higher yield than using shorter time. So, 90 min of reaction time gave a good result than other reaction times used here. In other words, the biodiesel yields increases with increasing the reaction time. However, based on the results, it shows that the biodiesel yields were lower when reaction time of 120 min was used. This undesirable result may be due to the higher soap formation when longer reaction time was used. Thus, the rate of soap formation was also increased. Conversion graph shown in (Figure 4).

Influence of molar ratio on biodiesel yield

Generally, the stoichiometry of the reaction requires three moles of methanol per mole of triglycerides to yield three moles of biodiesel and one mole of glycerol. Methanol is a commonly used alcohol for transesterification because of its low price and highly reactive nature. In this experiment, cottonseed oil were used with varying molar ratios of methanol and oil (4:1,5:1,6:1,7:1,8:1) and 1.0 wt% N_aOH at 60°C with 90 min reaction time. As mentioned, the transesterification activity also depends on the molar concentrations of methanol to oil and also associated with the type of catalyst used. If the molar ratio increased from 4:1 to 6:1 and then biodiesel yield content also

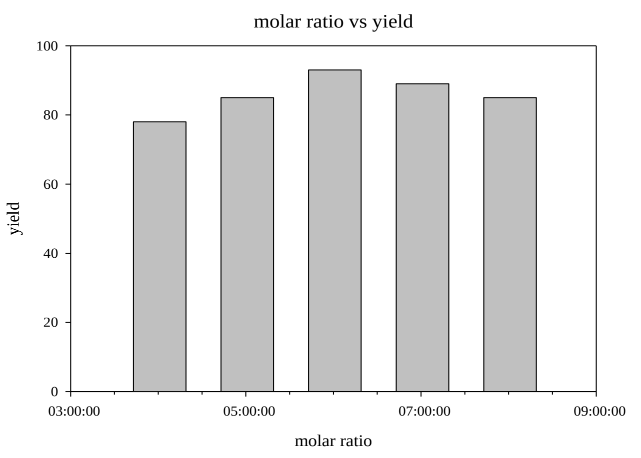

Figure 5: Biodiesel yield with molar ratio.

increased for cottonseed oil. Now, molar ratio increases from 6:1 to 8:1, methyl esters content decreased biodiesel yield and excess of molar ratio increases much then biodiesel yield also increases. Excess of methanol is required to shift the equilibrium favorably during transesterification for better yields of biodiesel. Therefore, 6:1 molar ratio gives a better result than other molar ratios and also repeated same procedure done for cottonseed oil. The higher alcohol molar ratio interferes with the separation of glycerol because there is an increase of solubility. In addition, an excess of alcohol was able to increase the conversion of di-monoglycerides, but there is possibility of recombination of esters and glycerol to form monoglycerides because their concentration and also increasing during the course of the reaction, in other words the reactions conducted with low molar ratios. Many researchers have reported an alcohol to oil molar ratio of 6:1 to be the optimal ratio, while reported that the maximum biodiesel production 93%was obtained at a molar ratio of 6:1 in transesterification of cotton seed oil. Conversion graph shown in (Figure 5).

Characterization of Cotton Seed Methyl Ester (Biodiesel)

Elementary analysis of biodiesel & oxygen and calorific value

In general, the calorific value (or heat of combustion) of methyl ester is less than that of fossil diesel due to presence of C-O and C-C bonds. The fossil diesel is made up of a mixture of various hydrocarbon molecules and contains little oxygen (up to 0.3%) and small amount of sulfur (0.25%), while the methyl ester consists of three basic elements namely: carbon, hydrogen, significant amount of oxygen and negligible amount of sulfur. The increase of O_2 in methyl ester (biodiesel) is related to the reduction of C and H, causes the lower value of lower calorific value (LCV) of Cotton seed methyl ester as compared to that of fossil diesel, because O_2 is ballast in fuel and 'C and H' are the sources of thermal energy. Therefore, the stoichiometric air-fuel ratio of Cotton seed methyl ester will be lower than fossil diesel. The Cotton seed methyl ester contains 9.46 % of O_2 as a result the combustion efficiency of the Cotton seed methyl ester will be increased [23].

Table 1: Elemental composition of cotton seed methyl ester.

Element (m%)	Petro-Diesel	CSOME	ASTM D6751-02
Carbon	86.25	70.96	77
Hydrogen	12.5	11.43	12
Nitrogen	0	1.31	----
Oxygen	0.25	9.46	0.05
Sulfur	1.0	0.15	11
C/H	6.9	6.20	----

Hydrocarbons

Hydrocarbons containing between six and ten carbon molecules are the top components of most fuels, regardless of whether they are alkanes, alkenes, or cyclic. In general, these molecules are burned to produce energy. The cotton seed methyl ester contains 70.96 % carbon and 11.43 % hydrogen as shown in (Table 1).

FFA analysis of biodiesel using GC

The fatty acid composition of the oils seems to have an important role in the performance of the biodiesel in diesel engines. Based on the fatty acid composition and many other parameters, the EU biodiesel specifications will be mandatory to limit the oxidative stability, as it may be a crucial parameter for injection pump performance. Moreover, the stability of the fuel is a quality parameter established by the ANP- National Petroleum Agency in Brazil, being its evaluation and control necessary.

Vegetable oils are natural products consisting of ester mixtures derived from glycerol (triglyceride), whose chains of fatty acid contain about 14 to 20 carbon atoms with different degrees of unsaturation. The transesterification reaction consists in the conversion of the

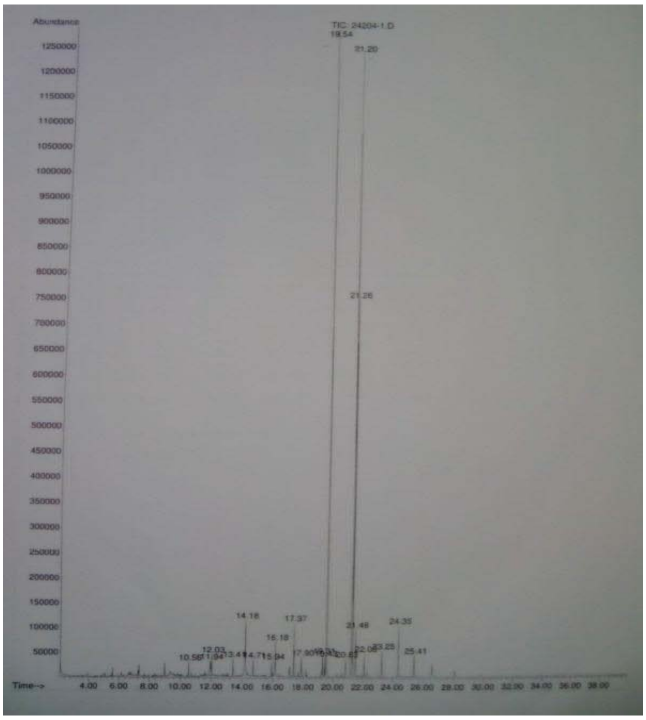

Figure 6: GC- 5973 N MSD Analysis for Methyl Ester of Cotton Seed Oil.

Table 2: Measured values of Fatty acids in the methyl ester of cotton seed biodiesel.

Fatty acids	Ratios	Methyl ester composition
Oleic	18:1	14.73
Linoleic	18:2	45.53
Stearic	18:0	2.06
Palmitic	16:0	30.43
Palmitoleate	16:1	1.03
Myristate	14:0	2.39
Others (Dodecane,Tridecane Tetradecane,Pentadecane, Hexadecane)		3.83

triglyceride molecules, by means of the action of short chain alcohol, i.e., methanol, ethanol into the corresponding fatty acid esters [24] (Figure 6).

The biodiesel is mainly formed by transesterification of saturated and monounsaturated fatty acids while the remaining polyunsaturated and some bulk saturated fatty acid are responsible for high viscosity of the biodiesel. The higher level of unsaturated fatty acid reduces fuel quality, because of its easy oxidation. As a rule saturated fatty acid such as 16:0 or 18:0 are stable than unsaturated ones like 18:1, 18:2 and 18:3 which decreases the fuel quality. The result also shows that methyl esters of biodiesel obtained from transesterification has more percentage of saturated fatty acids with less percentage of unsaturated fatty acids. The presence of saturated fatty acid in the obtained biodiesel leads to high viscosity, high cetane number and better biodiesel stability. The measured values of fatty acids present in the methyl ester of biodiesel are given in (Table 2).

Characteristics of Cottonseed Methyl Ester

The physical and chemical properties (Density, Lower calorific value and cetane number) of cotton seed methyl ester were calculated from the fatty acid composition obtained by GC. Properties of the methyl ester were tabulated in (Table 3) as follows.

Density

Density influences the efficiency of the fuel atomization for airless combustion system. It has some effect on the break-up of fuel injected into the cylinder. In addition, more fuel is injected by mass as the fuel density increases. The density of cottonseed methyl ester is 895 kg/m3 which meets the ASTM Standards.

Kinematic viscosity

It is defined as the resistance of liquid to flow and is the most important fuel features. This factor affects the operation of fuel injection, blending formation and combustion process. The high viscosity interferes with the injection process and leads to insufficient atomization. The kinematic viscosity of cottonseed methyl ester is 5.8 cSt which meets ASTM Standards.

Flash point

It is the minimum temperature of the fuel at which the fuel gives flash when it comes to contact with testing flame. It is an important parameter from the safety point of view such as safe for transport, handling, storage purpose and safety of any fuels. This is higher than petrol diesel which has flash point of 70°C. A fuel with high flash point may cause carbon deposits in the combustion chamber.

Table 3: Comparison of Physico-chemical properties of CSOME with ASTM standards.

Property	Petro Diesel	CSOME	ASTM D6751-02 (Biodiesel)
Carbon chain (Cn)	HC C_8-C_{32}	FAME C_{14}-C_{20}	FAME C_{12} – C_{22}
Density @30 °C D93(kg/m³)	825	895	870-900
Lower calorific value(Kj/kg)	42500	38500	-----
Kinematic viscosity@40 °C D445(cSt)	2.25	5.8	1.9-6.0
Cetane Number D613	49	52	47 min.
Iodine Value DIN5324 1(g Iodine/100g)	38	95	120 max
Flash point, Closed cup (°C) D93	70	162	130 min
Fire point(°C)	98	184	--------
Pour point(°C)	-20	-5	-15 to 16

Cetane number

Measure of the ignition quality of diesel fuel; higher this number the easier it is to start a standard (direct-injection) diesel engine. It denotes the percentage (by volume) of cetane (chemical name Hexadecane) in a combustible mixture (containing cetane and 1-Methylnapthalene) whose ignition characteristics match those of the diesel fuel being tested.

Lower calorific value

It is a measure of the energy produced when the fuel is burnt completely which also determines the suitability of Methyl ester as an alternative to diesel fuel. The calorific value of Methyl ester is normally lower than diesel due to oxygen content of Methyl ester.

Iodine value

Iodine value or iodine number is defined as the number of grams of iodine taken up by 100 g of oil or fat. In this case, addition reaction takes place across the double bonds of unsaturated fatty acids present in the fat by the addition of a halogen, such as iodine. Thus, the iodine number gives the indication of the degree of unsaturation of fats. The average number of double bonds can then be calculated from the halogen consumption, the double bonds in oil sample are halogenated using Wij's solution (iodine monochloride). Excess iodine monochloride is reduced to free iodine in the presence of potassium iodide. The amount of free iodine is then determined by titrating the solution with sodium thiosulphate using starch as indicator.

Molecular weight

The average molecular weight of the cottonseed methyl ester is calculated by considering the weight percent of each fatty acid and their corresponding molecular weights.

These values are meeting ASTM D 6751-02 standards, so this biodiesel suitable for direct usage in Diesel engines physical and chemical properties of cotton seed biodiesel.

Conclusion

This study revealed that biodiesel could be produced successfully from the cottonseed oil by alkali-catalyzed transesterification. The production of biodiesel from cotton seed oil offers a triple-facet solution: economic, environmental. In considering the range of tests performed in this project, the specifications used to identify acceptable biodiesels do not need any additions. The viscosity of cotton seed oil reduces substantially after transesterification and is comparable to petro-diesel and the physical and chemical properties of biodiesel produced conform to EN/ASTM standards .The effects of different parameters such as reaction time, temperature, catalyst concentration and reactant ratio on the biodiesel yield were analyzed. We can conclude that using water as a washing agent does not affect the reaction productivity at the same time it increases purity of the product. The unique variables that affect the biodiesel production are the catalyst concentration and the molar ratio alcohol/oil. 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. The elemental composition of biodiesel is less than Diesel. Consequently, Biodiesel emits less pollutant than diesel.

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