

Optimization of Effective Parameters of Bio-Diesels Extracted From Cotton Seed Oil, Palm Oil, Sunflower Oil & Coconut Oil

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Abstract—Bio fuels, fuels derived from biomass have been gaining the attention as of highly renewable, biodegradable and locally available. Biodiesel, obtained from vegetable oil or animal fats and Bio crude, synthetic oil. Bio fuels are carbon-neutral, nontoxic and reduce emission of volatile organic compounds. These fuels are not only green in nature but also help to reduce dependence on imported oil. Vegetable oil is a promising alternative fuel for CI engine because it is renewable, environment friendly and can be produced in rural areas. This article is comparative study of use of mineral diesel & bio diesel derived from cotton seed oil, palm oil, sunflower oil & coconut oil and also reports experimental data on production of fatty acid methyl ester from cotton seed oil, palm oil, sunflower oil & coconut oil using KOH as a catalyst. The variables, reaction temperature, reaction time, catalyst concentration & oil to alcohol (methanol) molar ratio affecting, yield & viscosity of bio diesel from these oil were optimized during transesterification process. The variable investigated reaction temperature (45°C-55°C), reaction time (20-40 min), catalyst (KOH) concentration (0.5-1.5 w/wt %), & oil to alcohol (methanol) molar ratio (10-20 w/wt %) & result obtained are graphically compared.

Keywords:- Biodiesel, Transesterification, FAME (Fatty Acid Methyl Ester) , FFA (Free Fatty Acid), Reaction temperature, Reaction time, Catalyst Concentration, Molar ratio.

I. INTRODUCTION

Biofuels offer an attractive option for meeting part of India's energy needs. Biofuels, in theory, can be produced from a wide variety of domestic feedstock. Like solar or wind power, biofuels are considered renewable energy sources as they rely on plant or waste products. This paper deals with biodiesel, a subset of biofuels that can substitute for petroleum diesel. The experience of some other countries in biodiesel production is encouraging. Global biodiesel production increased six fold between 2004 and 2008, from 2 billion liters to more than 12 billion liters. The European Union (EU) contributed more than two-thirds of this production. The top producers in the EU were Germany, France, Italy, and Spain. Besides the EU, the main biodiesel producers were the United States, Argentina, Brazil, and Thailand. Brazil introduced mandatory biodiesel blending of 2% in January 2008 and set a target of 5% by 2013. It started blending 3% in July 2008, and increased the blend to 4% in July 2009 and 5% in January 2010. Bio fuels are affordable substitutes for imported fossil fuels, generate rural income and employment, and reduce GHG emissions. Bio-diesel is an alternative to petroleum-based fuels derived from vegetable oils, animal fats, and used waste cooking oil including triglycerides. Vegetable oils are widely available

from various sources, and the glycerides present in the oils can be considered as a viable alternative for diesel fuel. They have good heating power and provide exhaust gas with almost no sulphur and aromatic polycyclic compounds. Vegetable oils are produced from plants, their burning leads to a complete recyclable carbon dioxide (CO₂). CO₂ associated with solar energy falling on earth gets converted in to the feedstock through photosynthesis. Vegetable oils available through this feedstock can be used to produce biodiesel. The use of vegetable oil for energy purposes is not new. It has been used world over as a source of energy for lighting and heating since time immemorial. The present availability of vegetable oils in the world is more than enough to meet the edible oil requirements, and surplus quantity available can partially meet requirements of biodiesel production. However, there is a considerable potential to further enhance the oilseeds production in the world to meet the increasing demand for food and biodiesel [1]. Biodiesel is an alternative fuel that can be used for reducing pollution to the air. It can be derived from vegetable oil like (mustard, soybean, rapeseed, canola, neem, jatropha etc) and animal fat (tallow) sources. is an environmental friendly fuel, which reduces the emission risks to the nature. It does not contain any sulphur, aromatic compounds and gives reduced soot. Due to its environmental and economic benefits, production of biodiesel is quickly adopted around the world [2]. Vegetable oil has high viscosity, that it cannot be used directly in conventional engines. Hence, its viscosity is lowered by the transesterification process. The molecular structure and the properties of the vegetable oil/animal fat are converted to methyl ester which is popularly known as Bio-diesel [3]. Air pollution is a major problem which induces the attention of biodiesel. Biodiesel is safer to breathe, nontoxic and biodegradable [4]. This can be used in conventional engines without or few modifications [5]. Internal combustion engine can use biodiesel as an alternative fuel which will emit reduced CO, HC and CO₂ [6]. In commercial processes, a catalyst is used to accelerate transesterification. After the reaction, the catalyst will be separated and the crude biodiesel needs a post-processing for further purification in order to meet the ASTM biodiesel standards. This separation/purification adds operating and capital cost to biodiesel production. The type of catalysts used depends on the nature of the feedstock. For example, homogeneous base catalysts such as KOH, NaOH, NaOCH₃, and KOCH₃ are usually used to accelerate the reaction for purified feedstock. The reaction occurs at moderate temperatures around 60°C and at atmospheric pressure with a short reaction time of around an hour [7]. This produces high biodiesel yield but requires a refined feedstock. To deal with the problems posed by impurities and post-process

separation, many alternative processes have been suggested. These include pre-treatment with an esterification reaction with an acid catalyst to lower FFAs [9], direct reactions with a heterogeneous catalyst for easier separation [8], reactions with enzymes [10] and reactions in supercritical alcohol. This last process is very interesting since the reaction is done without a catalyst and is less sensitive to water and free fatty acid than conventional homogeneous catalyst. Moreover, supercritical alcohol reactions reach complete conversion within a very short time, around 4 minutes, and the product yield remains high. The process does, however have one major drawback, it must occur under very severe conditions consisting of high temperature and pressure, which might lead to high energy consumption [11].

II. ABOUT SUNFLOWER OIL, COTTON SEED OIL, PALM OIL & COCONUT OIL:

We are confining our study to optimization & comparison of process parameters of bio-diesels extracted from sunflower oil, cotton seed oil, palm oil & coconut oil.

A. Sunflower oil: Sunflower oil is the non-volatile oil compressed from sunflower (*Helianthus annuus*) seeds. Sunflower oil is commonly used in food as frying oil, in cosmetic formulations as an emollient. The world's largest sunflower oil producers now are Ukraine, Russia and Argentina. The oil content of the seed ranges from 22 to 36% (average, 28%); the kernel contains 45–55% oil [12].

B. Cotton Seed Oil : Cottonseed oil is a cooking oil extracted from the seeds of cotton plants of various species, mainly *Gossypium hirsutum* and *Gossypium herbaceum*, that are grown for cotton fiber, animal feed, and oil. Cotton seed has a similar structure to other oilseeds such as sunflower seed, having an oil-bearing kernel surrounded by a hard outer hull; in processing, the oil is extracted from the kernel. Its fatty acid profile generally consists of 70% unsaturated fatty acids (18% monounsaturated, and 52% polyunsaturated), 26% saturated fatty acids. When it is fully hydrogenated, its profile is 94% saturated fat and 2% unsaturated fatty acids (1.5% monounsaturated, and 0.5% polyunsaturated) [13].

C. Palm oil: Palm oil is an edible vegetable oil derived from the mesocarp (reddish pulp) of the fruit of the oil palm, primarily the African oil palm and to a lesser extent from the American oil palm and the maripa palm. Palm oil is naturally reddish in color because of a high beta-carotene content. It is not to be confused with palm kernel oil derived from the kernel of the same fruit, or coconut oil derived from the kernel of the coconut palm (*Cocos nucifera*). The differences are in color (raw palm kernel oil lacks carotenoids and is not red), and in saturated fat content: Palm mesocarp oil is 41% saturated, while Palm Kernel oil and Coconut oil are 81% and 86% saturated respectively [14].

D. Coconut Oil: Coconut oil is an edible oil extracted from the kernel or meat of matured coconuts harvested from the coconut palm (*Cocos nucifera*). It has various applications in food, medicine, and industry. Because of its high saturated fat content it is slow to oxidize and, thus, resistant

to acidification, lasting up to two years without spoiling. Many health organizations advise against the consumption of high amounts of coconut oil due to its high levels of saturated fat. Coconut oil was once prevalent in western countries like the United States. With a long shelf life and a melting point of 76 degrees, coconut oil was a favourite in the banking industry. But a negative campaign against saturated fats in general, and coconut oil in particular, led to most food manufacturers abandoning coconut oil in recent years in favour of hydrogenated polyunsaturated oils that come from the main government-subsidized cash crops in the US, particularly corn and soy. These hydrogenated oils contain Trans fatty acids [15].

III. EXPERIMENTAL SETUP AND MATERIALS

A 500 ml was used as a reactor. The beaker was placed on magnetic stirrer whose temperature could be controlled. A separating funnel was used for settling and separating purpose. Sunflower oil, cotton seed oil, palm oil & coconut oil purchased from local market. All reagents for transesterification reaction i.e., catalysts, methanol, ethanol, NaOH used from chemistry lab chemistry department of GGI, Ambala, Haryana.

IV. TITRATION

A. Free Fatty Acid Calculation

Equipment used: Flask, Phenolphthalein indicator, Ethanol, Sunflower oil, cotton seed oil, palm oil & coconut oil as per your choice, Hot plate, Burette, Sodium Hydroxide (NaOH). Procedure

B. Free Fatty Acid (FFA) Content

Vegetable oil typically contains from 2 percent to 6 percent free fatty acids. Free fatty acid levels will increase with the amount of time vegetable oil has been heated. The presence of too high level of free fatty acids will retard or stop the transesterification reaction. To ensure a successful conversion to bio-diesel, determining the exact amount of catalyst needed to neutralize the acids by performing a titration test is worthwhile. Adding too much catalyst will result in excessive amounts of soap in the final bio-diesel product. If too little catalyst is added, transesterification will not occur.

Chemicals required for FFA estimation were described below:

1. Phenolphthalein indicator.
2. Ethanol & NaOH Sol.

Phenolphthalein indicator was prepared by adding 0.5 gm of phenolphthalein pellets to 50 ml of distilled water & 50 ml of ethanol. NaOH sol was prepared by adding 0.4 gm of NaOH Pellet in 100 ml distilled water. The method for FFA estimation was described below:

- Take 10 ml Vegetable (Sunflower) oil sample & 50 ml of Ethanol.
- Mix them in a conical flask and heat this sample until bubbling starts when bubble formation starts suspend the further heating and immediately add 2-3 drops of phenolphthalein indicator.
- Now, using the titration flask, start adding NaOH of 0.1 normality drop wise in above sample till the colour of whole sample becomes uniformly same (pink).

- When the colours of whole sample becomes uniformly same, and then stop the further addition of NaOH in above sample of oil.
- Note the amount of NaOH consumed during this process.
- Use the Standard formulae to find the FFA content of oil sample

$$\text{FFA} = (\text{N} \times 28.2 \times \text{V}) / 10$$

Where,

V = Volume of NaOH consumed in titration.

N = Normality of NaOH.

Table 1 FFA contents for Sunflower oil, Cottonseed oil, Palm oil & cottonseed oil

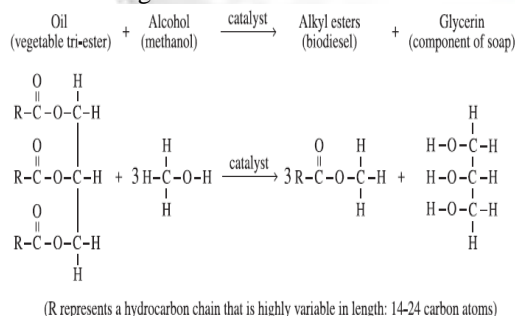
S.No.	Oil Taken	Free Fatty Acid%
1	Sunflower oil	0.14
2	Cottonseed Oil	1.78
3	Palm Oil	0.17
4	Coconut oil	0.31

V. TRANSESTERIFICATION

The process of converting vegetable oil into bio-diesel is called transesterification, a chemical process that removes the glycerin stem from the molecule, resulting in a much smaller molecule, called an ester, which improves its characteristics for use as an engine fuel. Methanol was commonly used as the alcohol in the transesterification process. A catalyst was required or the conversion process would not occur. The two most common catalysts used in producing bio diesel were potassium hydroxide and sodium hydroxide. Most biodiesel recipes recommend the amount of catalyst be 0.5 percent to 1.5 percent of the total amount of oil in the batch.

A. Procedure for transesterification:

1. Take 160 gm of Vegetable (Sunflower) oil in a conical flask.
2. Preheat this oil at 60 °C for 30 min in water bath shaker.
3. In a separate flask mix 0.5% of KOH (catalyst) & 20% of methanol by weight of Oil.
4. Add this solution to preheated oil sample.
5. Maintain the above sample at 55 °C for 20 min at constant stirring in a water bath shaker.



Vegetable oil + Methanol + potassium hydroxide
→ Bio Diesel Fuel + Glycerin

Fig.1: Basic Transesterification reaction

6. After this put the samples in separating funnel for 24 hrs so that glycerin will settle to the bottom and can be drained off.
7. The liquid left in separating funnel is crude bio-diesel as it contains alcohol (Methanol) and catalyst (KOH) in it.
8. Now boil the bio-diesel to get pure, moisture free bio-diesel ready to be used in the engine.
9. Same procedure will be repeated for other oil cottonseed oil, palm oil & coconut oil shown in fig 1.

Table 2. Different apparatus and standards used for fuel characterization

S.No	Name of fuel property	Methods/Standards
1	Kinematic Viscosity	Redwood Viscometer, IS: 1448 [P:25]:1976
5	FFA Content (%)	Titration with 0.1 N NaOH

VI. DESIGN OF EXPERIMENT FOR THE OPTIMIZATION OF TRANSESTERIFICATION OF SUNFLOWER OIL, COTTON SEED OIL, PALM OIL & COCONUT OIL.

A. Effect of various Parameters on Yield & Viscosity

The variables Catalyst Concentration, Reaction Temperature Reaction Time and Methanol-Oil –Ratio on Yield is studied from research papers & basis on the highest yield & low viscosity obtained the reaction temperature was varied from 45°C to 55°C, the catalyst concentration was varied from 0.5 to 1.5 w/wt%, the reaction time was varied from 20 to 40 minutes & the methanol-oil-ratio was varied from 10-20 w/ wt%. The graph is plotted for different oil namely sunflower oil, cottonseed oil, palm oil & coconut oil are shown in fig 2 & fig 3.

- Y1 is yield for reaction time 20 min, reaction temperature 45° C, catalyst concentration 0.5 w/wt% and methanol-oil-ratio 20 w/wt%
- Y2 is yield for reaction time 30 min, reaction temperature 50° C catalyst concentration 1.0 w/wt% and methanol-oil-ratio 10 w/wt%
- Y3 is yield for reaction time 40 min, reaction temperature 55° C, catalyst concentration 1.5 w/wt% and methanol-oil-ratio 15 w/wt%.

B. Procedure for calculating viscosity :

Viscosity can be defined as the resistance to flow of liquid due the internal friction between the liquid and surface. It plays an important role in the performance of an engine fuel system operating through a wide range of temperature.

Table 3. Yield % & Viscosity From Variables Reaction Time, Reaction Temperature, Catalyst Concentration & Alcohol %

S. No.	Reaction Time (min)	Reaction Temperature (°C)	Catalyst Concentration (gm)	Alcohol %	Yield%	Viscosity (mm ² /s)
SUNFLOWER OIL						
1	20	45	0.5	20	97.30	5.42
2	30	50	1.0	10	88.80	5.76
3	40	55	1.5	15	86.58	5.90
COTTONSEED OIL						
1	20	45	0.5	20	98.01	4.15
2	30	50	1.0	10	85.36	4.79
3	40	55	1.5	15	84.60	4.56
PALM OIL						
1	20	45	0.5	20	96.39	5.96
2	30	50	1.0	10	89.99	5.84
3	40	55	1.5	15	83.84	5.45
COCONUT OIL						
1	20	45	0.5	20	94.54	6.55
2	30	50	1.0	10	89.95	5.31
3	40	55	1.5	15	85.71	4.98

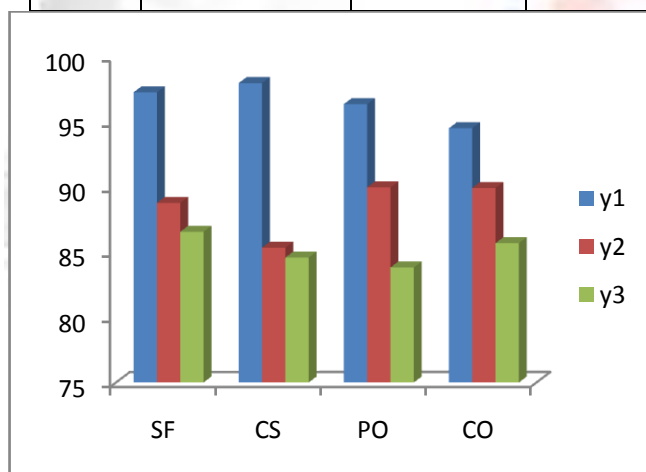


Fig. 2: Value of Yield on Process parameters

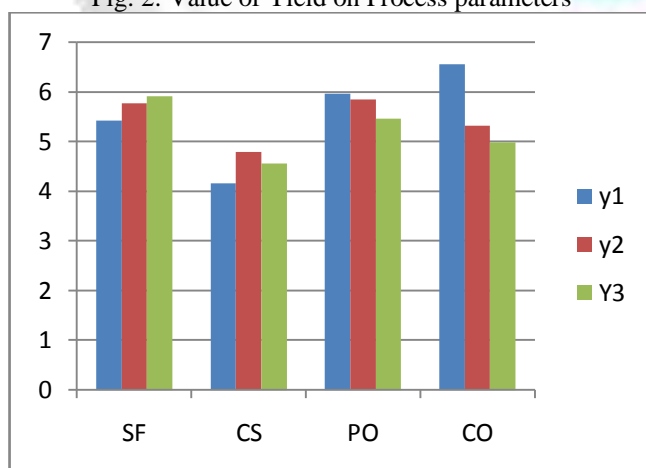


Fig. 3: Value of Viscosity on Process parameters

Kinematic viscosity affects the injection system. Low viscosity can result in an excessive wear in injection pumps and power loss due to pump leakage whereas high viscosity

may result in excessive pump resistance, filter blockage, high pressure and coarse atomization and fuel delivery rates. A Redwood viscometer was used for measurement of kinematic viscosity of selected fuel samples. The instrument measures the time of gravity flow in seconds of a fixed volume of the fluid (50 ml) through specified orifice made in a agate piece as per IS:1448 [P:25] 1976. The apparatus could be used for flow time between 30 to 2000 seconds. The fuel was filled in a cup fitted with agate jet at the bottom up to a specified level indicated in a cup. The cup was surrounded by water jacket having an immersion heater. The heater was heated to 40°C by regulating the rate of heating using a voltage regulator of the instrument. A simple metallic ball was used to open and close the agate jet. A standard 50 ml volumetric glass was kept below the agate jet to collect a falling fuel samples & viscosity obtained are shown in table 3. Each test was replicated thrice. Kinematic viscosity in centistokes was then calculated from time units by using the relationships:-
 $V_k = 0.26 t - 179/t$, When $34 < t < 100$ and
 $V_k = 0.24 t - 50/t$, When $t > 100$
 Where, V_k = Kinematic viscosity in centistokes, mm²/s, t = Time for flow of 50 ml sample.

VII. RESULT & DISCUSSIONS:

A. Characteristics

The overall studies based on the production of biodiesel from sunflower oil, cotton seed oil, palm oil and coconut oil, fuel characterization of sunflower oil, cotton seed oil, palm oil and coconut oil were carried out. The recovery of esters by transesterification of the above mentioned oils with methanol are affected by varying the composition of catalyst & the following conclusions can be drawn:

- In Sunflower Methyl Ester: The recovery of sunflower methyl ester at lowest kinematic viscosity (5.42 mm²/s)

was 97.30 % from 160 gram is possible at the following standardized concentration of catalyst.

- In Cottonseed Methyl Ester: The recovery of cotton seed methyl ester at lowest kinematic viscosity (4.15 mm²/s) was 98.01% from 160 gram is possible at the following standardized concentration of catalyst.
- In Palm Methyl Ester: The recovery of palm oil methyl ester at lowest kinematic viscosity (5.45 mm²/s) was 83.84 % from 160 gram is possible at the following standardized concentration of catalyst.
- In Coconut Methyl Ester: The recovery of coconut seed methyl ester at lowest kinematic viscosity (4.98 mm²/s) was 85.71% from 160 gram is possible at the following standardized concentration of catalyst.

B. FFA Content of Methyl Esters :

The FFA contents of Sunflower oil (0.14) and Palm oil (0.17) were observed less than the Coconut oil (0.31) and Cotton seed oil (1.78). If the oil has a high water or free fatty acid (FFA) content the reaction will be unsuccessful due to saponification (saponification is defined as the reaction of an ester with a metallic base and water) commonly known as making soap, and make separation of the glycerol difficult at the end of the reaction. The FFA content of the raw oil will determine the quantity of biodiesel as the final product. A very low content of FFA (<0.2) can give a full 100% yield.

C. Kinematic Viscosity of Methyl Esters :

The kinematic viscosity of Sunflower, Cottonseed, Palm and Coconut methyl ester were found as 5.42, 4.15, 5.45 and 4.98 mm²/s respectively at 40° C. Coconut methyl have higher kinematic viscosity as compared to others. Sunflower, cottonseed & palm ester had the kinematic viscosity in range of petroleum diesel (1.6-6 mm²/s as per ASTM).

VIII. CONCLUSION

In this study, the optimum parameters for high percentage of yield and low kinematic viscosity was selected by varying the varying parameters reaction temperature, reaction time, molar ratio of Alcohol & Catalyst concentration for Sunflower oil, cotton seed oil, palm oil & coconut oil.

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