Synthesis and Stability Characteristics of Sesame Methyl Ester

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Abstract— As the Fossil fuels are declining day by day and the demand of fuel from alternative resources is increasing rapidly, Biodiesel is one of best Alternatives to the petroleum fuel. Acid-catalyzed biodiesel production process is a competitive alternative for biodiesel production using edible Oils as raw materials and is less complex than alkali-catalyzed approaches. So, Sesame Biodiesel was synthesized from Sesame Oil and Stability Characteristics were studied. The Results showed that the Biodiesel made from Sesame Oil can be used as an alternative to the conventional Petroleum Diesel Fuel. So, this paper is the Experimental Study on the Synthesis and Stability Characteristics of Sesame Methyl Ester Synthesized from Sesame Oil**.**

Keywords—*Sesame Biodiesel, Biodiesel, Stability Characteristics, Sesame Oil, Biodiesel, Sesame Methyl Ester*

I. INTRODUCTION

As the Fossil fuel is declining day by day, the demand of fuel from alternative resources is increasing rapidly. Due to Increase in Demand of Fuels, the price of fuels is also increasing day by day. So, various researches are being carried out to find the best alternative of fussil fuelsBiodiesel is one of Alternative fuel. Acid-catalyzed biodiesel production processes is a good alternative for biodiesel production using waste cooking oil as raw materials and are less complex than alkali-catalyzed approaches. [1]. Biodiesel is defined as monoalkyl ester derivatives of long chain fatty acids that are produced through the Transesterification of various oils with alcohol and alkaline catalysts .

Biodiesel is best *alternative* to the conventional petroleum fuel. Itis made by mono-alkyl-esters of long chain fatty acids derived from vegetable oils or animal fat [2]. We can Synthesize biodiesel from available vegetable oils like palm, soybean, peanut, sunflower, rape, coconut, karanja, neem, cotton, mustard, Jatropha, linseed and castor through a chemical process known as transesterification [3-5] It is commercially produced through the transesterification of various types of oils like vegetable oils, used frying oils and many other varieties of Oils [6]. Biodiesel is obtained by the chain reactions of oil and methanol or ethanol in the presence of an catalyst like KOH, NaOH or H_2SO_4 [7]. There is a large number of vegetable oil that are being used as an alternative to biodiesel in various countries like soybean oil is being used in U.S.A., palm oil in Malaysia and Indonesia, rapeseed and sunflower oil in Europe and coconut oil is being used widely in Philippines [8]. Biodiesel as an alternative fuel can be used in various diesel engines in pure form (B100) or it can be blended with petroleum diesel in various concentrations and Ratios like B10, B20, B30, B40, B50, B60, B70, B80 and B90 [9].

Biodiesel is biodegradable in Nature and environment friendly fuel but due to the reason that it is unstable in nature and can loss its quality and properties over time, thus Oxidation stability is most necessary parameter of biodiesel. The oxidation stability of biodiesel is always lower than that of petroleum-based diesel. Fatty wastes in oil cause more oxidation because they vary in level of unsaturation. This leads to generation of more carbon-carbon double bonds and fewer hydrogen molecules on the fatty acid chains. When Biodiesel is kept in presence of Oxygen, It easily got oxidized as the oxygen is rapidly attached to the alkyl group in Biodiesel. [10]. So, Oxidation Stability plays an important role in the quality of Biodiesel.. Thus Mustard Biodiesel from Waste Mustard oil was synthesized and its Oxidation Stability was checked by using Professional Biodiesel Rancimat 893. So, below is the full paper representing the various methods and conclusions.

II. MATERIALS

Sesame oil is used for Transesterification reaction so that biodiesel can be synthesized from this oil. This oil is used for many cooking processes and domestic use. The alcohol used in this experiment is Methanol and a Catalyst used is Potassium hydroxide This KOH used in this experiment is in solid state i.e. in form of pallets. For the determination of acid number, Isopropyl alcohol was used in which Phenolphthalein was used as an indicator in the titration.

III. METHODS

Alkyl esters are produced when vegetable oil is combined with alcohol in presence of catalyst. The fatty acids of vegetable oil exchange places with the (OH) groups of the alcohol producing glycerol and methyl, ethyl or butyl fatty acids ester depending on the type of alcohol used [14]. The alcohol used is mainly 15% of mass of oil and catalyst used is 1-2%of mass of oil respectively in this process. As alcohol methanol or ethanol is used whereas potassium hydroxide (KOH) is used as catalyst [15].

Table 1. Physico-Chemical Properties and Standards of Biodiesel in Accordance with ASTM D-6751, EN-14214, and IS-15607 Standards: [12][13]

Property	ASTM D 6751 test method	ASTM D 6751 limits	EN 14214 test method	EN 14214 limits	IS 15607 test method	IS 15607 limits
flash point (C)	D-93	min.130	EN ISO 3679	min. 120	IS 1448 P:21	min. 120
viscosity at 40 C (cSt)	D-445	1.9-6.0	EN ISO 3104	3.5-5.0	IS 1448 P:25	2.5-6.0
sulfated ash (% mass	D-874	max. 0.02	EN ISO 3987	Max. 0.02	IS 1448 P:4	Max. 0.02
sulfur (% mass)	D-5453/ D-4294	max. 0.0015 (S 15) max. 0.05 (S 500)	EN ISO 20846/20884	Max. 0.0010	ASTM D 5453	Max. 0.005
copper corrosion	D-130	max. 37	EN ISO 2160	max. 1	IS 1448 P:15	max. 1
Cetane number	D-613	min. 47	EN ISO 5165	min. 51	IS 1448 P:9	min. 51
Water and sediment (vol. %)	(vol. %) D-2709	max. 0.05			D-2709	Max. 0.05
Conradson carbon residue (CCR) 100% (% mass)	D-4530	Max. 0.05	EN ISO 10370	Max. 0.3	D-4530	Max. 0.05
Neutralization value (mg, KOH/g)	D-664	Max. 0.50	EN ISO 14104	Max. 0.5	IS 1448 P:1/sec:1	Max. 0.50
free glycerin (% mass)	D-6584	max. 0.02	EN ISO 14105/14106	max. 0.02	D-6584	Max. 0.02
total glycerin (% mass	D-6584	max. 0.24	EN ISO 14105	max. 0.25	D-6584	max. 0.25
phosphorus (% mass)	D-4951	max. 0.001	EN 14107	max. 0.0010	D-4951	max. 0.001
distillation temperature	D-1160	90% at 360 C			not under spec.	min 90%
oxidation stability at 110 C (h)	EN 14112	Min. 3 hr.	EN ISO 14112	Min 6 Hr.	EN 14112	Min. 6 h.
CFPP(°C)	D-6371		EN 116	Variable	IS 1448 P1:10	

A. Free Fatty Acids

FFA (Free Fatty Acid) of any vegetable oil plays important role in synthesis of biodiesel. In Transesterification reaction, the higher value of free fatty acids can cause retardation. To ensure a successful conversion to bio-diesel, determining the exact amount of catalyst needed to neutralize KOH/gm 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. If there will be greater value of free fatty acids , the lesser will be desired product. The free fatty acid number must lie 1 to 2.5. For sesame oil the free fatty acid value was 2.0. To Find Acid number in vegetable oil, pour 2 to 3 gram of Oil in a beaker and Add 50 ml Isopropyl Alcohol. After it, pour potassium hydroxide Normality N/10 solution in the burette. Now add 3-5 drops of Phenolphthalein indicator and titrate it against KOH till the color changes to pink. Note Amount of KOH used. And by applying the Formula of Acid number, we can easily find out FFA of that Oil. In general FFA= 2 X ACID NUMBER.

B. Transesterification Process

For synthesis of biodiesel the process is Transesterification is widely used. In this process, when alcohol is added with oil in the presence of catalyst, the formation of Alkyl ester and Glycerin takes place. Alkyl esters are desired product and is considered as biodiesel whereas glycerin is waste product but from this formation of can take place. At beginning the oil is heated at temperature of 60°C. [17]Meanwhile alcohol is heated with catalyst till their perfect solution is made. After getting desired temperature of oil (60°C) the catalyst and alcohol solution is poured in oil and this solution is heated for one hour. The following reaction takes place

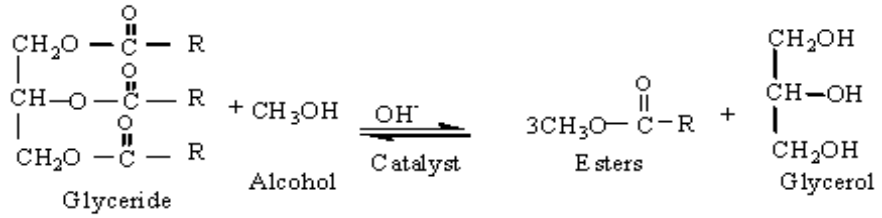


Fig.1: Transesterification Reaction

C. Separation

After completion of 12 hours, the alkyl esters and glycerin get separated and glycerin can be separated by downward tap. The Alkyl esters are preserved for further use.

Fig. 2. Separation of sesame ethyl esters and glycerin

D. Washing Process

Process of washing methyl esters is very important for synthesis of biodiesel. Water is used to remove the impurities and foreign particles Water is used for washing because water has very high tendency to combine with the impurities and other particles which may create problem to the alkyl esters. In this process water is heated to 45°C and is mixed with the Alkyl ester in separation flask. After 2 hours, the impurities are entrapped with water and this water becomes milky. The milky water is removed by the exit tap of separation flask. This process continuous till the transparent water is obtained.

Fig.3. Washing Process

E. Heating

In the previous process of washing**,** some of water drops get mixed with alkyl esters. To remove those water drops ans excess ethanol present, the process of heating is done. As we know that the boiling point of Water is 100°C and that of Methanol or Ethanol is 60°C. So, Alkyl esters are heated and Stirred at the Temperature of 110°C so that to remove all unwanted particles available in Biodiesel. After Heating, the Liquid obtained is called Biodiesel and can be used for further studies.

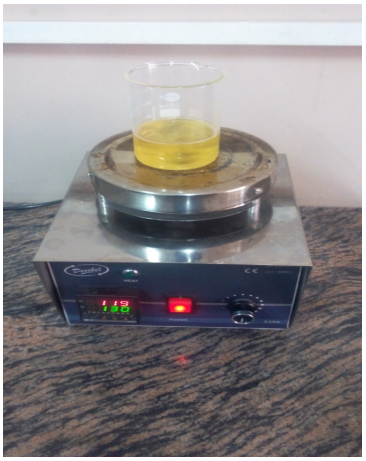


Fig.4. Heating alkyl esters at 110°C

IV. EXPRIMENTAL SECTION

A. Materials and Method

The sesame oil was taken. The oil was poured in a beaker and heated to 60°C by using digital heater. In the meantime 1.5% KOH and 15% methanol was poured in other beaker and stirred to make it perfect solution. After this the solution was poured in oil and heated at constant temperature of 60°C for one hour. After one hour this solution was poured in separation flask for 12 hours undisturbed. The separation of SME (Sesame methyl Esters) and Glycerin takes place. After which glycerin and methyl esters were separated.

The water at 45°C was poured in methyl esters for 2-3 hours for washing process. This process was repeated for 5-6 times in order to remove all the impurities. The obtained methyl esters were heated at 110°C in order to evaporate the excess methanol and water from the solution.

Thus after series of these processes we get the final product as sesame methyl esters which is our required sesame biodiesel.

B. Biodiesel Stability Testing

Storing a biodiesel is a big problem because it gets oxidized easily. So oxidation stability of freshly prepared sesame biodiesel was checked using electric instrument called Profession Biodiesel Rancimat, which is the most efficient device to check oxidation stability of a biodiesel. Oxidation Stability of biodiesel was studied in the Rancimat equipment model 893 (Metrohm, Switzerland), according to EN-14112

specifications [17]. The Biodiesel made from used mustard Oil was tested and it showed effective oxidation stability of 5.48 hours.

Fig.5. Rancimat equipment model 893 (Metrohm, Switzerland)

In the method of Rancimat, steam of air at the rate of 10L/h is passed through biodiesel sample by keeping temperature 110°C. The vapours released during the oxidation process, together with the air, are passed into the flask containing 50 mL of triple deionized water. The electrode present to measure conductivity. The electrode is connected to a measuring and recording device. It indicates the end of IP when the conductivity begins to increase rapidly. This is due to the rapid dissociation of voltaic carboxylic acid produced during process of oxidation and absorbed in water. When the conductivity of this measuring solution is recorded continuously, an oxidation curve is obtained whose point of f inflection is known as the IP or oil stability index. The Principle Diagram of Rancimat is as followed:

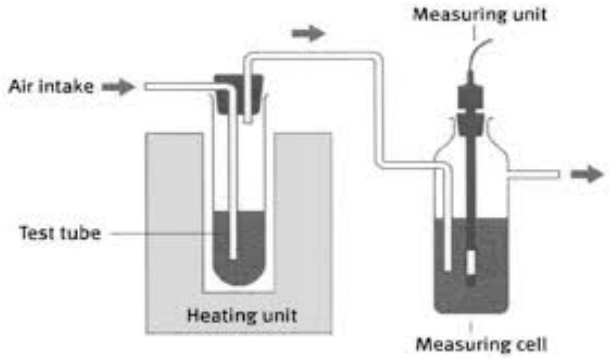
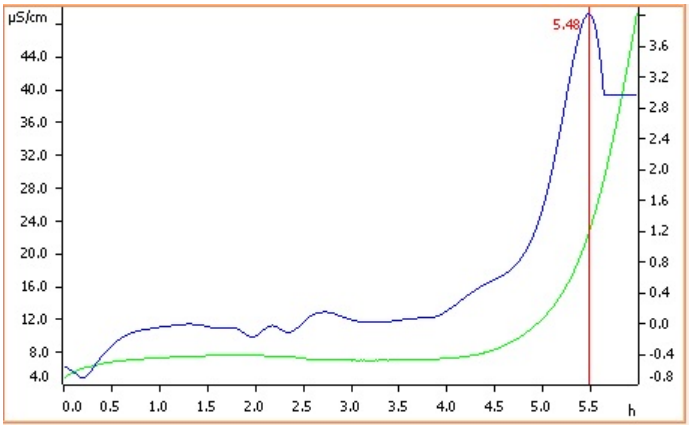


Fig.6. Principles of Measurement of the Rancimat Test Method (EN-14112/IS-15607)

3 gram sample of sesame biodiesel was poured into test tube and Rancimat was started for results of oxidation stability. After 5 hours, the sesame biodiesel was oxidized. Below the graph obtained from Stabnet Software of Professional Biodiesel Rancimat Showing the Oxidation Stability.

V. RESULT

The Sesame Methyl Ester was synthesized from sesame oil and its Stability Characteristics were Studied using Professional Biodiesel Rancimat 893. After the Processes of Rancimat, the Time versus Conductivity graph is obtained, which shows that the Oxidation Stability of Sesame Biodiesel derived from sesame oil is 5.48 Hours. This shows that the Sesame Biodiesel exhibits good Oxidation Stability as per as Oxidation of biodiesel is concerned. The oxidation stability of Sesame Methyl Ester can be increased by using different natural or artificial Antioxidants.



Graph 1: The graph showing Oxidation Stability of Sesame Biodiesel as 5.48 h.

Result Definitions:

- Induction Time: 5.48 hours
- Standard Time: 19635.54 hours
- Result Standards (IND): 5.478254352465923

VI. CONCLUSION

In this experiment, the Sesame oil was used as feedstock and Sesame Methyl ester was obtained by the Transesterification of Sesame Oil. The Oxidation Stability of Sesame Biodiesel was tested in Professional Biodiesel Rancimat and it is concluded that:

- Sesame Biodiesel Showed the Oxidation Stability of 5.48 hours respectively.
- The Oxidation Stability of Sesame Methyl Ester can be increased by using Artificial Antioxidants Like TBHQ, TBHT, PG, PY, BHT and BHQ.
- This experiment also motivates to use natural resources which are less expensive, easy available and Environment Friendly.

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