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Synthesis and Oxidation Stability Characteristics of Sesame and Mustard Methyl Esters in Blended Form (B50)

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Abstract: ***As the amount of fossil fuels is decreasing rapidly, we need to rely on latest technologies to conserve fossil fuels and discover alternative sources of energy. One of the alternative source of renewable energy is 'Biodiesel'. It is defined as mono-alkyl ester derivatives of long chain fatty acids which can be used as an alternative to diesel. Thus, biodiesel was synthesized from sesame oil and mustard oil by trans-esterification process respectively. These two biodiesels were blended (B50). Oxidation Stability (OS) of biodiesel tells about the quality of synthesized biodiesel and it was checked in professional biodiesel Rancimat 893 according to IS 15607. Biodiesel synthesized from Sesame and Mustard oil has showed an effective OS in blended form (B50). The OS of Sesame and Mustard Methyl Ester(SSME) has come to be 5.48 hours respectively. So, this research paper is the Experimental Study on the Synthesis and Oxidation Stability Characteristics of Sesame and Mustard Methyl Ester in blended form (B50) Synthesized from Sesame and Mustard Oil.***
Keywords: Biodiesel, Sesame Oil, Diesel, Renewable Energy, Sesame Methyl Ester (SME); Mustard Methyl Ester (MME); Oxidation Stability; Transesterification reaction; Methyl Esters (ME).

I. INTRODUCTION

As the demand of Fossil fuel is hiking every day, we need to depend on the alternative sources of energy. Biodiesel is one of those alternative fuels. Acid-catalyzed biodiesel production processes, a viable alternative for biodiesel production, uses cooking oil as raw material and are less complex than alkali-catalyzed approaches [1] . On one hand the consumption of fossil fuels is increasing day by day and on the other hand the quantity of fossil fuel is also decreasing rapidly. By using fossil fuels, not only we are degrading the environment but also putting a financial burden on the economy of our country. Biodiesel is alternative to the conventional petroleum fuel and is made by mono-alkyl-esters of long chain fatty acids derived from edible oils or non-edible oils [2]. Biodiesel can be synthesized from available edible oils like sesame, palm, soybean, peanut, coconut, sunflower, rape seed, neem, cotton, mustard, jatropha, linseed and castor through a chemical process known as trans-esterification [3-5] Biodiesel can be produced commercially by the process of trans-esterification using various types of edible and non-edible oils [6]. Biodiesel is obtained by the chain reactions of oil and methanol or ethanol in the presence of catalysts like KOH, NaOH, H2SO4 etc. [7]. There are many edible oils like soya-bean, palm, coconut, sunflower that are being widely used in various countries like U.S.A., Malaysia, Indonesia, Philippines [8]. Biodiesel, 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 eco-friendly fuel. Because it is unstable in nature and can lose its quality and properties over time, Oxidation stability is most necessary parameter for biodiesel. Fatty wastes in oil causes more oxidation because there is a variation in the level of unsaturation. This leads to formation of more carbon-carbon double bonds and fewer hydrogen molecules on the fatty acid chains. In presence of oxygen, Biodiesel gets easily oxidized because the oxygen rapidly attaches to the alkyl group in Biodiesel [10]. So, Oxidation Stability plays an important role in the quality of Biodiesel. OS of SME & MME was checked by using Professional Biodiesel Rancimat 893 in blended form. The oxidation stability of biodiesel is always lower than that of petroleum-based diesel [11]. So, below is the full paper representing the various methods and conclusions.

II. MATERIALS

Sesame seeds are imported specially from Madurai (South India). The oil is extracted from these seeds by the process of grinding. The Mustard oil was extracted from Mustard. Then the extracted oil is used for trans-esterification reaction so that ME can be

obtained. Sesame oil is used for many purposes like cooking, massage etc. The chemicals used in this experiment are Methanol and the Catalyst used is Potassium hydroxide. The KOH used in this experiment is in solid state i.e. pallets. Isopropyl alcohol was used in which Phenolphthalein was used as an indicator in the titration for the determination of acid number.

III. METHODS

There is formation of esters, when vegetable oil combines with alcohol in the presence of a catalyst, the fatty acids of vegetable oil exchange places with the (OH) groups of the alcohol and produces glycerol and ME when methanol is used. The concentration of catalyst can affect the rate of formation of ME. Its excess quantity can lead to soap formation. [12].The Catalyst used may vary from 1% to 2% of Mass of oil and the Alcohol used in this process is mainly 15 % of mass of Oil respectively. Mainly, potassium Hydroxide is used as catalyst. NaOH can also be used but According to some scientific reasons of effect of Na ions on Biodiesel i.e. why KOH is used. Methanol or Ethanol is used as Alcohols [13].

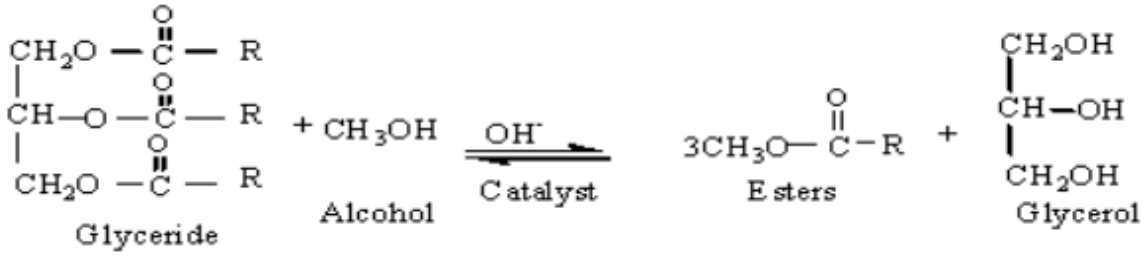
Property	ASTM D 6751 test method	ASTM D 6751 limits	EN 14214 test method	EN 14214 limits	IS 15607 test method	IS 15607 limits	SSME (B50)
flash point (C)	D-93	min.130	EN ISO 3679	min. 120	IS 1448 P:21	min. 120	145
viscosity at 40 C (cSt)	D-445	1.9-6.0	EN ISO 3104	3.5-5.0	IS 1448 P:25	2.5-6.0	4.70
sulfated ash (% mass)	D-874	max. 0.02	EN ISO 3987	Max. 0.02	IS 1448 P:4	Max. 0.02	0.014
sulfur (% mass)	D-5453/ D-4294	max. 0.0015 (S 15)	EN ISO 20846/20884	Max. 0.0010	ASTM D 5453	Max. 0.005	0.0035
copper corrosion	D-130	max. 37	EN ISO 2160	max. 1	IS 1448 P:15	max. 1	0.93
Cetane number	D-613	min. 47	EN ISO 5165	min. 51	IS 1448 P:9	min. 51	55.6
Water and sediment (vol. %)	(vol. %) D-2709	max. 0.05			D-2709	Max. 0.05	0.30
Conradson carbon residue (CCR) 100% (% mass)	D-4530	Max. 0.05	EN ISO 10370	Max. 0.3	D-4530	Max. 0.05	0.39
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	0.32

free glycerine (% mass)	D-6584	max. 0.02	EN ISO 14105/14106	max. 0.02	D-6584	Max. 0.02	0.01
total glycerine (% mass	D-6584	max. 0.24	EN ISO 14105	max. 0.25	D-6584	max. 0.25	0.13
phosphorus (% mass)	D-4951	max. 0.001	EN 14107	max. 0.0010	D-4951	max. 0.001	<0.001
distillation temperature	D-1160	90% at 360 C			not under spec.	min 90%	>90%
OS at 110 C (h)	EN 14112	Min. 3 hr.	EN ISO 14112	Min 6 Hr.	EN 14112	Min. 6 h.	1.77 h

Table 1. Physico-Chemical Properties and Standards of Biodiesel in Accordance with ASTM D- 6751, EN-14214, and IS-15607 Standards: [14][15]

A. Transesretification Process

The Trans-esterification process is widely used for the synthesis of biodiesel from sesame oil. In this process, when sesame oil is mixed with methanol in the presence of KOH, the formation of ME and Glycerin takes place. The desired product is ME and is considered as Biodiesel. Glycerin is the waste product but it can be used to make soap [16]. So, firstly the sesame oil is heated to 60°C temperature. In the Meantime, methanol is mixed with KOH (catalyst) till the perfect solution is made. After achieving the desired temperature, the methanol and KOH solution is poured into the heated sesame oil and is left for one hour with continuous steering at constant temperature of 60°C. The same process is done in case of Mustard oil. The following reaction takes place:



Reaction during Transesterification Process

After this continuous steering for 1 hour, the Mixture is poured into separation flask and allowed to settle for 12 hours. After the period of 12 hours, ME and glycerin will get separated in case of both sesame and mustard.

B. Sepration

After the Settle time of 12 hours, the ME and glycerin get separated and the glycerin is removed from the separation flask from the downward Tap. The ME is preserved for further processes. The glycerin is a component of soap and can be used in soap formation by various methods.

C. Washing Process

Mainly, water washing is used to remove tiny particles and impurities from the ME. Water has very high tendency to combine with the impurities which may cause hindrance in formation of ME i.e. why it is used for washing. The water is heated to 45°C and is mixed with the Alkyl ester in separation flask. After 2 hours water becomes milky because the impurities get entrapped with water. The milky water is removed by the exit tap of separation flask. This process continuous till the transparent water is obtained at the bottom of separation flask. The Water Wash is most important process in the synthesis of biodiesel.

D. Heating Process

Some drops of water get mixed with ME. The ME are heated at 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 of Stability and other Engine Studies.

IV. EXPERIMENTAL SECTION

A. Material And Methods

Sesame seeds are imported specially from Madurai (South India). The oil is extracted from these seeds by the process of grinding. The Mustard oil was extracted from Mustard. Then Mustard Oil and sesame oil were poured in a Beaker and was heated to 60°C by using Digital Heater and Magnetic Stirrer respectively. In the Meantime, 1.5% KOH was mixed in 15% methanol and the Solution was poured into the Beaker. After then, the whole Solution was supposed to be at constant temperature and continuous steering for One hour. After continuous steering for one hour, the solution was poured into Separation flask and the solution was not disturbed for 12 hours so that ME and glycerin settled to their respective places. After 12 hours, the glycerin was removed from the exit tap of Separation flask and the remaining ME was kept in separation flask for further processes. The obtained MME and SME was washed with heated water (45°C) four times till the transparent water was not obtained. After this Process of 6 hours, The MME & SME was poured into a beaker and was again heated at the temperature of 110°C so that to remove water and methanol from the MME& SME respectively. Thus, after the series of processes, The Mustard Biodiesel made from Mustard Oil and Sesame Biodiesel made from sesame Oil were obtained as a final product

B. Biodiesel Blending

The samples of Sesame Biodiesel and Mustard Biodiesel are Blended in B50 i.e. The sesame biodiesel and mustard biodiesel are taken in same proportion (25 grams each) and blended by simply mixing each other at room temperature. As a result, we get blended sesame methyl esters and mustard methyl esters namely Sesame and Mustard Methyl Esters (SMME).

C. Biodiesel Stability Testing

We cannot store biodiesel for long time as it gets oxidized easily. Thus, an electrical instrument called Professional Biodiesel Rancimat is used to check the Oxidation stability of every freshly synthesized biodiesel. This is most efficient device to check the oxidation stability of every Biodiesel. According to EN-14112, IS 15607, ASTM D 6751 specifications, Oxidation Stability of biodiesel was studied in the Rancimat equipment model 893 (Metrohm, Switzerland).[17]. The Biodiesel synthesized from Sesame Oil was tested and it showed an effective Oxidation Stability of 5.48 Hours. The Rancimat method is also called the automated swift test and automated version of the previously used and extremely complicated and time consuming Active Oxygen Method. In the Rancimat Method, the oxidation is induced by passing a stream of air at the rate of 10 L/h through biodiesel sample (3 g), kept at constant temperature 110°C. The vapors released during the oxidation process, together with the air, are passed into the flask containing 50 mL of triple Deionized water, and contain an electrode for measuring the 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 accelerated increase is caused by the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water. When the conductivity of this measuring solution is recorded continuously, an oxidation curve is obtained whose point of inflection is known as the IP or oil stability index. The Principle Diagram of Rancimat is as followed: -

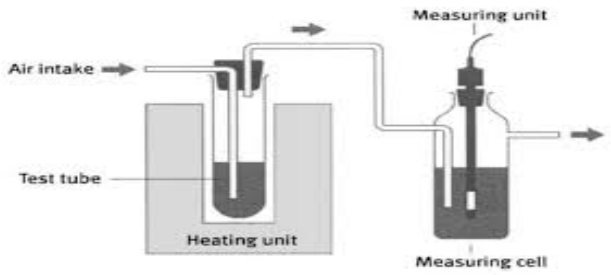


Fig.1. Principles of Measurement of the Rancimat Test Method (EN- 14112/IS-15607)

The 3g sample of Mustard ME was poured into test tube and the apparatus was started for the results of Oxidation Stability of MME. After the time period of 6 hours, the Mustard Biodiesel was oxidized in the Professional Biodiesel Rancimat. Below is the graph obtained from Stabnet Software of Professional Biodiesel Rancimat Showing the Oxidation Stability.

V. RESULTS AND CONCLUSION

The results obtained by this method, Sesame Biodiesel, showed the positive results as Oxidation Stability is came to be 5.48 Hours. So, this OS is fulfilling the three major Standards as Europe Standard, American Standard and Indian Standard using the 14112 standards, the Rancimat method. If the Oxidation stability will be more, The Efficiency and Quality will also be good. As, the Oxidation stability of Mustard ME is 5.48 h, the prepared sample meets the requirements of IS 14112 Standard of Biodiesel. So, Mustard Biodiesel can be used as an alternative to the Petroleum Diesel as it fulfils the ASTM D 6751 limits of Oxidation Stability of minimum 3 hours. But it not satisfies Indian Standards and European standards for Oxidation Stability of Minimum 6 Hours. Thus, by using Antioxidants like Tert-butylated hydroxytoluene (TBHT), tert-butylated phenol derivative (TBP), tert butyl hydroxquinone (TBHQ)[18].

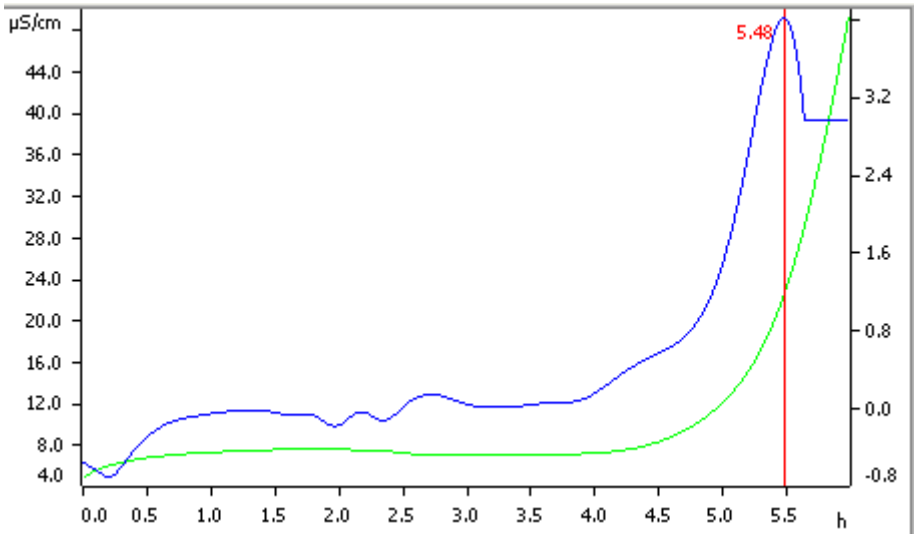


Fig.2.Oxidation Stability of SMME(B50) is 5.48 hours

VI. RESULT DEFINITIONS

A. Induction Time: - 5.48 h
B. Standard Time: - 19635.54 h
C. Induction Time(IND): - 5.5 h

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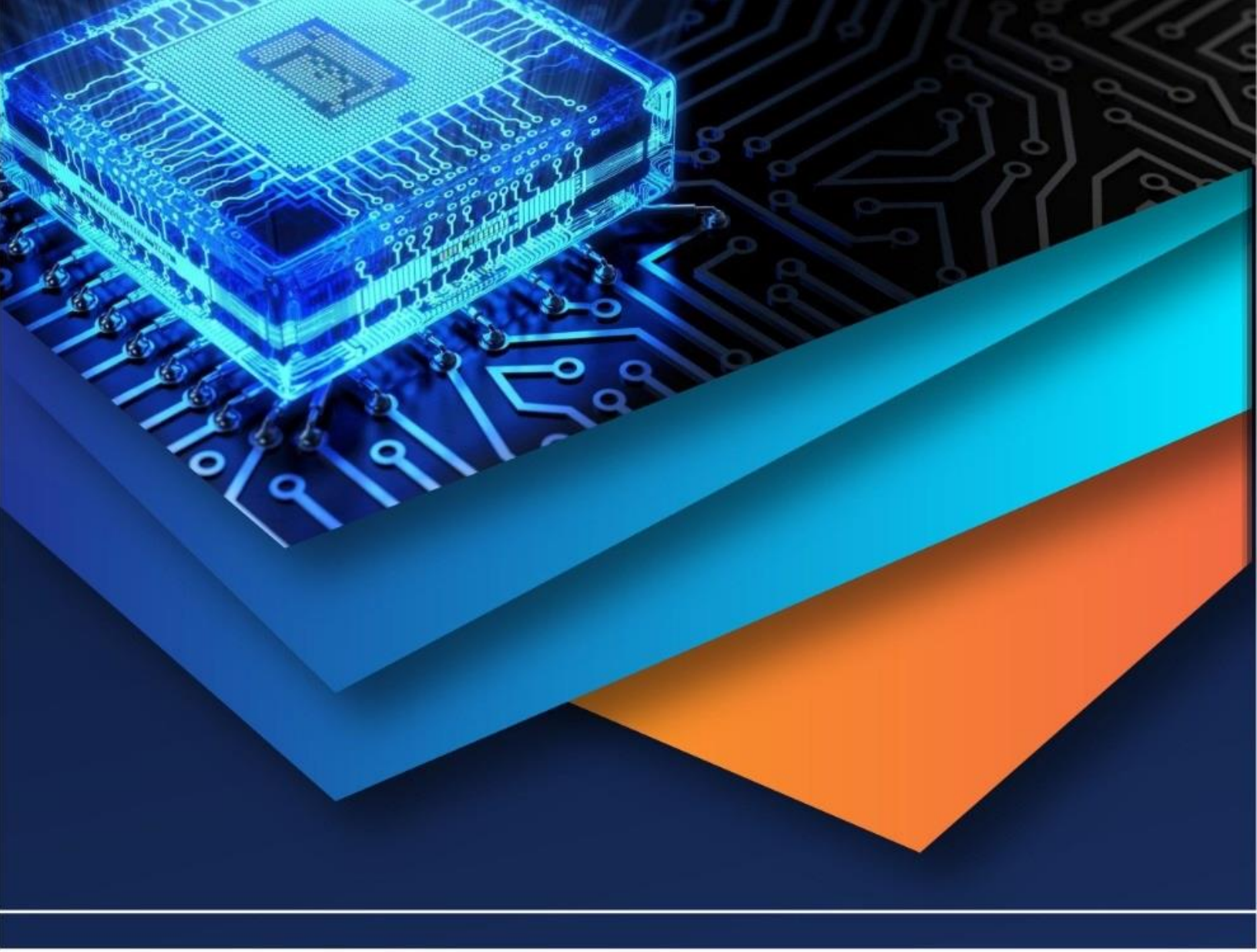

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