International Journal on Emerging Technologies **5**(1): 99-106(2014)

ISSN No. (Online) : 2249-3255

Effective Process Parameters of Mustard Oil Biodiesel - A Review and Analysis

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(Received 19 March, 2014 Accepted 17 May, 2014)

ABSTRACT: The world consumption of fuels is undoubtedly unstable causing world economic crisis, the worst compared to other economic recession that took place at different era. By using biodiesel, the problem could be tackled. 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. Thus, present study relates bio-diesel used in experimental work was prepared from Mustard oil by the process known as transesterification. The transesterification of Mustard oil with alcohols, in the presence of base catalyst potassium hydroxide (KOH) and methanol as solvent, by means of single step batch transesterification process in order to obtain biodiesel fuel was studied. The reaction has been done in water bath stirrer. The catalyst concentrations which affect the product yield during transesterification process were investigated experimentally to optimize it. The parameters such as reaction temperature, reaction time, Catalyst Concentration & Alcohol % were optimized & analyzed & graphs are plotted against Viscosity & Yield obtained.

Keywords: Ester, FFA (Free Fatty Acid), FAME (Fatty Acid Methyl Ester), Catalyst, KOH, NaOH

I. INTRODUCTION

The humankind is increasingly approach towards high energy crisis, on account of greater energy consumption than its supply. The demand is increasing further because of the world becoming more developed in new technologies. Plentiful and economical energy is the lifeblood of current nation. The world presently meets the energy requirements majorly from fossil fuels. Over the last few decades, the world has experienced an alarming increase in consumption of fossil fuels such as coal, oil, natural gas, etc. Fossil fuels are hydrocarbon deposits formed inside the earth's crust by the decay of plant and animal matter over long periods of time extending up to millions of years. They are non-renewable sources of energy. Today's majority of industrial as well as household activities are accomplished by using energy derived from these fossil fuels. The growth in world energy consumption in 2007 was 2 % per year and a growth rate of 1.1% per year is expected in the future [1]. Another concern regarding the use of fossil fuels is their detrimental effects on the environment. Large amounts of particulate matter, sulfur, and green house gases, such as carbon dioxide and carbon monoxide, are constantly released into the atmosphere by burning fossil fuels. These gases pollute the atmosphere and cause green house effects that ultimately lead to global warming. A solution to this problem is renewable energy resources also known as alternate fuels or non-conventional sources of energy. Alternate fuels refer to substances that have two characteristics similar to fossil fuels and can efficiently replace fossil fuels. Examples of renewable fuels include biodiesel produced from vegetable oils and ethanol produced from plant biomass [2].

Sadeghinezhad *et al* studied that in the wake of oil crisis, the world is looking for the alternative source of energy where bio-diesel came into play as an attractive renewable alternative fuel. However, it was realized that extensive utilization of bio-fuel would tax the food chain and could lead to food shortages. So, the use of a blend of bi-fuel with conventional fuel was suggested to balance its usage which still could provide beneficial greenhouse effect. In the hot and cold climate bio-diesel cannot conveniently replace fossil fuel but in the controlled environment with modified combustion equipment, bio-diesel can be used as an alternate fuel. Having lower heating value, bio-diesel is consumed more in comparison to the fossil diesel fuel. Bio-diesel also generates more NO_x emission, which is an adverse environmental pollutant. The raw material source of bio-diesel limits food growing ground which is ultimately becoming a great concern. A dilemma of using bio-diesel as an alternative for mineral fuel has raised a concern about environment, engine performance and involved costs these have to be investigated in depth to provide a recommendation. Biodiesel is produced from renewable sources and it can play increasingly a major role in support of meeting energy demand in transportation systems although there have been in consistent trends for the performances of biodiesel engine and different range of gases emission during varied biodiesel blends and operating conditions or driving cycles [3].

Sadeghinezhad *et al* concluded that Biodiesel is produced from renewable sources and it can play increasingly a major role in support of meeting energy demand in transportation systems although there have been in consistent trends for the performances of biodiesel engine and different range of gases emission during varied biodiesel blends and operating conditions or driving cycles. Pressures on international grain marketing have influenced on prices during the past years. Due to the retarded grain growth and rapid growing demand for grains the price of biodiesel can be easily reversed. The global food economy is facing demand for food, feed, and fuel and also the future challenges of increasing land-use pressures and climatic changes. The agricultural productivity will have to grow significantly faster in the future than it had been in the recent past years. Lack of easy access to food will influence food prices, such as possible long-term illness, irreversible consequences for health, productivity, and well-being particularly if higher prices result in reduced food consumption by infants and preschool children If the current bio-fuel expansion continues, calorie availability in developing countries is expected to grow slowly which will lead to higher number of malnourished children, even though agricultural income would also accelerate [4].

Titipong Issariyakul *et al* determined that Biodiesel is gaining acceptance in the market as fuel and lubricant. It is expected that biodiesel industries will rapidly grow worldwide in the coming years and information on biodiesel feedstock, production, and characteristics will be crucial than ever especially for those using vegetable oils as feedstock as these are currently the major sources for making biodiesel. In the present paper, a comprehensive review is reported on feedstock, production technologies, and characteristics of biodiesel. More specifically, selected available vegetable oils are explored as feedstock for biodiesel production. Finally, biodiesel characteristics and parameters influencing the corresponding properties are revealed. Since this paper covers a wide range in biodiesel area, it serves as a general public education medium as well as a research reference for biodiesel production from vegetable oils. Therefore, selection of vegetable oil and production technology is vital for growth in biodiesel industries. In order to make an effective decision, in-depth information and understandings on biodiesel from vegetable oils is essential [5].

II. EXPERIMENTAL SETUP AND MATERIALS

The Mustard oil used in this present study was bought from Kiryana Shop of Kurukshetra, Haryana. All chemicals (Methanol, Ethanol, Phenopthaline, KOH Catalyst, and NaOH) were procured during experimentation from chemistry lab of Ambala college of Engineering and Applied Research, India. Water bath Stirrer was used for transesterification of Mustard oil. All chemicals (Methanol, Ethanol, Phenopthaline, KOH Catalyst, and NaOH) were procured during experimentation from chemistry lab of college. Numbers of sample bottles were purchased from Sadar Bazar, Ambala Cantt to put different biodiesel sample during experimentation. Water bath was used for transesterification of Mustard oil. The fuel properties have been determined by using equipments such as, Redwood viscometer. Some properties of Mustard oil biodiesel were determined at Bharat Test House, Rai, Sonepat (Haryana).The Mustard oil bio-diesel used in this present study was prepared at Ambala College of Engineering and Applied Research**,** Mithapur, Ambala Cantt.

In this process the ester was produced when vegetable oil combines with a simple alcohol in presence of a 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. The concentration of catalyst affects the level of ester recovery and requires experimental optimization as concentration of catalyst is less than required value reaction will not complete and if it is in excess quantity then saponification takes place which leads to soap formation so it is necessary to optimize catalyst concentration. The effect of process parameter were studied to standardize the transesterification process for estimating recovery of maximum ester yield as well as recovering ester of lowest possible viscosity. In order to standardize the catalyst concentration, three levels of catalyst (KOH) concentration (0.5%, 1.0%, and 1.5%) was set and the reaction time of 20 minutes, 40 minutes & 60 minutes and reaction temperature of 45˚C, 55˚C & 60˚C were taken from research papers.

III. TITRATION

A. Free Fatty Acid Calculation

Equipment used: Flask, Phenolphthalein indicator, Ethanol, Mustard oil , Hot plate, Burette, Sodium Hydroxide (NaOH).

(i) Free Fatty Acid (FFA) Content: Mustard oil typically contains from 2 percent to 5 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. Phenolphthalein indicator was prepared by adding 0.5 gm of phenolphthalein pallets to 50 ml of distilled water & 50 ml of ethanol. NaOH sol was prepared by adding 0.4 gm of NaOH Pallet in 100 ml distilled water.

The method for FFA estimation was described below:

1. Take 10 ml Mustard oil sample & 50 ml of Ethanol.
2. 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.

3. 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).
4. When the colours of whole sample becomes uniformly same, and then stop the further addition of NaOH in above sample of oil.
5. Note the amount of NaOH consumed during this process.
6. Use the Standard formulae to find the FFA content of oil sample

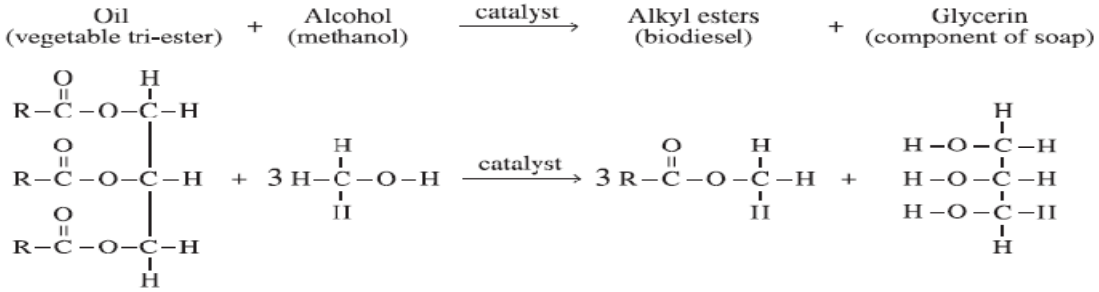
FFA = (N × 28.2 × V) / 10

Where,

V = Volume of NaOH consumed in titration.
N = Normality of NaOH.

IV. TRANSESTERIFICATION

Biodiesel is made from vegetable and animal oils. It can be used directly in diesel vehicles or blended with traditional petroleum diesel. The biodiesel manufacturing process converts oils and fats into chemicals called long-chain mono alkyl esters, or biodiesel. These chemicals are also referred to as fatty acid methyl esters (FAME) and the process is referred to as transesterification as show in Fig 1.



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

Fig. 1. Basic Transesterification reaction [13].

Fig. 2 provides a simplified diagram of the transesterification process. Roughly speaking, 100 pounds of Mustard oil or fat or other oils are reacted with 10 pounds of a short-chain alcohol (usually methanol) in the presence of a catalyst (usually sodium hydroxide [NaOH] or potassium hydroxide [KOH]) to form 100 pounds of biodiesel and 10 pounds of glycerin. Glycerin is a sugar, and is a co product of the biodiesel process as shown in Fig. 3.

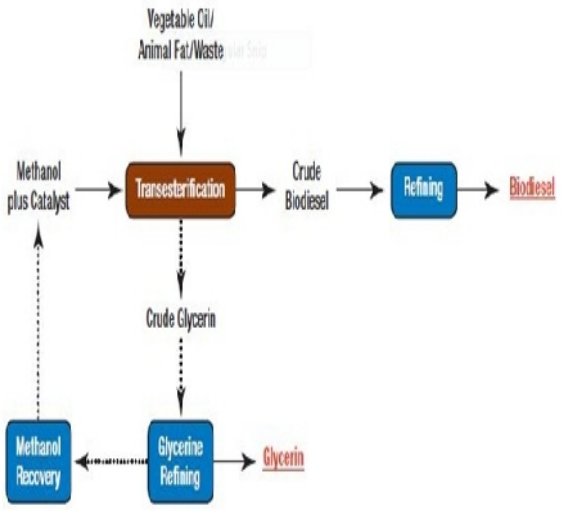


Fig. 2. Basic Process of Bio Diesel [13].

Fig. 3. Mustard Oil Bio Diesel with Glycerin.

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, $NaOCH_3$, and $KOCH_3$ 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 [6]. 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 [7], direct reactions with a heterogeneous catalyst for easier separation [8], reactions with enzymes [9] and reactions in supercritical alcohol [10]. 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.

A. Benefits of using biodiesel

1. Biodiesel has many environmentally beneficial properties. Biodiesel is commonly described as "carbon neutral." Burning biodiesel emits carbon dioxide (CO_2), but this emission is offset by the fact that the crop used to produce it uses CO_2 from the atmosphere to grow. (However, some CO_2 is released during the production of the fertilizer required to fertilize the fields in which the oil crops are grown.)
2. Biodiesel is rapidly biodegradable and completely nontoxic, meaning spillages represent far less of a risk than fossil diesel spillages.
3. Biodiesel has a higher flash point than fossil diesel and so is safer for storage or in the event of an accident.
4. Compared with fossil diesel, biodiesel combustion emits fewer pollutants such as carbon monoxide (CO) and particulates. Sulfur is almost completely eliminated. Nitrous oxides may stay the same or increase but can be reduced with a catalytic converter and/or by altering the engine timing.
5. Biodiesel is more lubricating than mineral diesel and so increases fuel injector pump life.
6. Biodiesel can reduce waste by recycling used oil.
7. Biodiesel has an energy balance of 3:1; i.e., it provides over three times the amount of energy used to produce it. [11].
8. Biodiesel can be used in the existing engines without any modifications. The use of biodiesel can extend the life span of diesel engines because it is more lubricating than petroleum diesel fuel. A lot of research work has already been carried out to use vegetable oil both in its pure form and also in modified form. Studies have shown that the usage of vegetable oils in pure form is possible but not preferable [12].

B. Advantages of using biodiesel

• It is renewable.
• It is energy efficient.
• It displaces petroleum-derived diesel fuel.
• It can be used as a 20% blend in most diesel equipment with no or only minor modifications.
• It can reduce global warming gas emissions.
• It can reduce tailpipe emissions, including air toxics.
• It is nontoxic, biodegradable, and suitable for sensitive environments [12].

C. Procedure for transesterification

As 27 samples are prepared & same procedure will be followed shown below in Table 1. Design of experiments.
1. Take 150 gm of Mustard oil in a conical flask.
2. Preheat this oil at 45 ˚C for 20 min in water bath shaker.
3. In a separate flask mix 0.5% of KOH (catalyst) & 10% of methanol by weight of Mustard Oil.
4. Add this solution to preheated oil sample.
5. Maintain the above sample at 45 ˚C for 20 min at constant stirring in a water bath shaker.
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. So, all the process will be repeated and yield and viscosity will be obtained.

Transesterification was done at 6:1 molar ratio and then allowed to settle for 24 hr in order to obtain maximum recovery of ester with lowest possible kinematic viscosity as reported by past researchers. Total 27 ester samples were prepared as shown in Table 1 to study the effect of the three levels of catalyst concentration on ester recovery and subsequent measure of their kinematic viscosity out of which 9 best are selected according to their yield & viscosity as shown in Fig. 4. The water bath for transesterification is shown in Fig. 5.

Table 1: Design of Experiments.

S. No	Reaction Temperature	Reaction time	Catalyst Conc.	Alcohol	Yield	Viscosity
1	45	20	0.5	10	98.60	4.62
2	45	20	0.5	10	98.35	4.63
3	45	20	0.5	10	98.56	4.64
4	45	40	1	15	84.69	4.86
5	45	40	1	15	84.63	4.87
6	45	40	1	15	84.88	4.85
7	45	60	1.5	20	89.98	4.79
8	45	60	1.5	20	89.13	4.79
9	45	60	1.5	20	88.99	4.78
10	55	20	1	20	99.38	4. 82
11	55	20	1	20	99.43	4.81
12	55	20	1	20	99.23	4.79
13	55	40	1.5	10	83.45	4.89
14	55	40	1.5	10	84.01	4.92
15	55	40	1.5	10	83.87	4.90
16	55	60	0.5	15	95.02	4.59
17	55	60	0.5	15	94.79	4.56
18	55	60	0.5	15	94.99	4.58
19	60	20	1.5	15	90.63	4.68
20	60	20	1.5	15	90.99	4.67
21	60	20	1.5	15	89.78	4.63
22	60	40	0.5	20	99.21	4.40
23	60	40	0.5	20	99.23	4.41
24	60	40	0.5	20	99.70	4.39
25	60	60	1	10	84.58	4.71
26	60	60	1	10	84.97	4.72
27	60	60	1	10	84.03	4.70

Fig. 4. Nine ester samples prepared.

Fig. 5. Wath bath shaker.

Table 2: Apparatus.

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

D. Kinematic Viscosity

Viscosity is defined as resistance to flow of liquid due to internal friction between the liquid and the surface. It plays an important role in the performance of the fuel system of engine operating through wide range of temperatures. It affects the fuel injection system as the 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, coarse atomization and fuel delivery rates. A Redwood viscometer (Table 2.) is used for measurement of Kinematic Viscosity of selected fuel samples. The instrument measures the time of gravity flow in seconds of fixed volume of the fluid (50ml) through specified orifice made in an agate piece as per IS: 1448 [P : 25] 1976.

The fluid was filled in a cup fitted with agate jet at bottom up to a specified level indicated in the cup. The cup was surrounded by water jacket having an immersion heater. The water was heated to 40˚C by regulating the rate of heating with the help of thermostat of instrument. A silver plated metallic ball was used to open and close the agate jet. A standard 50ml flask was kept below agate to collect falling fluid sample. Each test was replicated thrice. Kinematic viscosity in centistokes was then calculated using time units by using relationships given by Guthrie (1960).

vk = 0.26 t -179 / t

When 34 < t <100 and **vk = 0.26 t -50 / t**

When t > 100 where,

vk = Kinematic viscosity in centistokes

t = Time for flow of 50 ml sample, sec

Table 3: Optimization of parameters during transesterification.

S.No	Sample weight, gm	Catalyst %	Reaction Time, min	Reaction Temperature, ˚C	Alcohol %	Yield %	Viscosity, mm^2/sec
1	**150**	**0.5**	**40**	**60**	**20**	**99.70**	**4.39**
2	**150**	**1.0**	**20**	**55**	**20**	**99. 38**	**4.82**
3	**150**	**0.5**	**20**	**45**	**10**	**98.6**	**4.62**

V. RESULTS AND DISCUSSION

The 27 experiments were done in order to determine the optimum quantity of catalyst which gives maximum yield and minimum viscosity of bio-diesel during transesterification process out of which best 3 selected with maximum yield & minimum viscosity Table 3 and the percent methyl ester recovered along with its viscosity by transesterification process carried out at different quantity of catalyst concentration and constant reaction temperature. It is evident from the Table 3 that the recovery of Mustard oil methyl esters observed at different catalyst concentration varied between 74% to 99.7 % and viscosity ranged between 4.39 to 4.82mm^2/sec. It was, therefore, seen that highest recovery of 99.7 % of methyl ester and lowest viscosity of 4.39 mm^2/sec was obtained at 0.5% catalyst concentration by weight of Mustard oil when Mustard oil was reacted with 20% of weight of oil methanol at 60˚C for 40 minute in presence of 0.5 percent KOH and then allowed to settle for 24 hr. After the reaction was over within the specified time period the mixture was poured in to a separating flask and kept overnight for settling. The upper layer was methyl ester (biodiesel) and lower dark layer was glycerol.

This upper layer of biodiesel was now separated and then boiled at 105˚C for 10 minutes to remove moisture from it. Now the pure biodiesel was ready for using in diesel engine. The effect of KOH concentration was studied in the range of 0.5 to 1.5% (weight of KOH/weight of oil). The reaction temperature was kept constant at 60˚C. The results for different concentration of catalyst to oil are shown in Table 3.

A. Analysis and Discussion through graphs

The optimal level of the process parameters is the level with the greatest value. So, the results obtained are Larger is better as we need bio diesel production at highest level.

A) Graphs are plotted for Reaction temperature, Reaction Time, Catalyst Concentration & Alcohol percentage w.r.t. yield obtained as shown in Fig. 6. Following results we obtained:

1. In Fig. 6 from 1st graph, we conclude that as Reaction temperature increases, yield also increased from 84% to 94 %. It is directly proportional to yield.
2. In 2nd graph, when reaction time is less yield is at highest (96% to 80%), when the time increases yield decreases (80% to 87%).

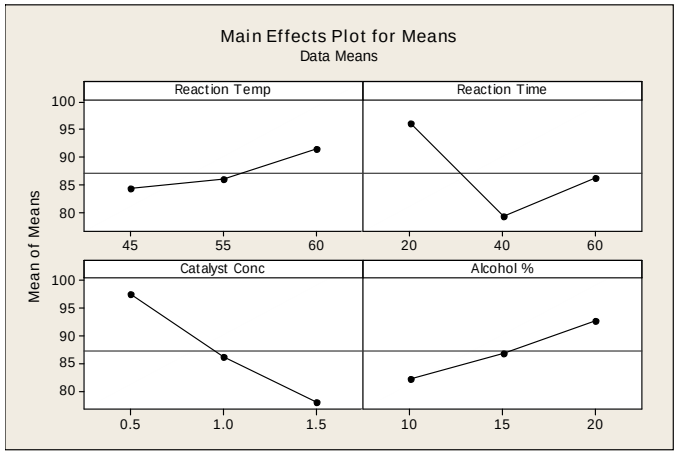


Fig. 6. Main Effects Plot for Means for Yield.

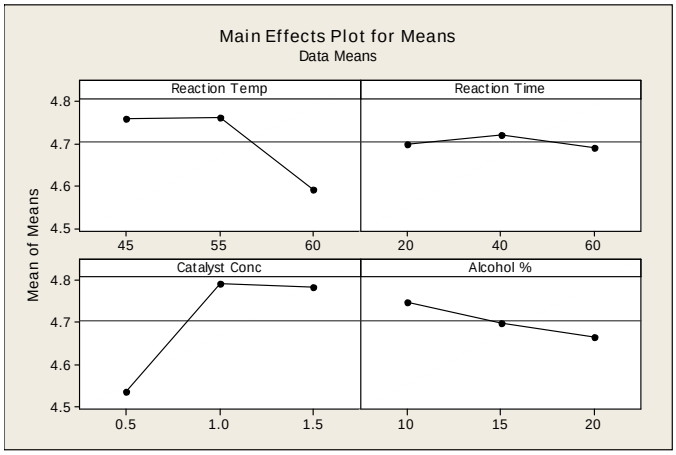


Fig. 7. Main Effects Plot for Means for Viscosity.

3. In 3rd graph, as catalyst concentration increases, yield decreases continuously from (99 % to 79%). It is inversely proportional to yield.
4. In 4th graph as Alcohol % increases, yield increases continuously (84% to 99.7%). It is directly proportional to yield.

So, it is conclude that catalyst concentration has maximum impact on yield

B) Graphs are plotted for Reaction temperature, Reaction Time, Catalyst Concentration & Alcohol percentage w.r.t. viscosity obtained as shown in 7. Following results we obtained:-

1. In Fig. 7 from 1st graph, we conclude that as Reaction temperature increases, Viscosity decreased. It is inversely proportional to viscosity.
2. In 2nd graph, when reaction time has very less effect on viscosity because increase in reaction time has less effect on viscosity.
3. In 3rd graph, as catalyst concentration increases, viscosity increases. It has direct impact on viscosity.
4. In 4th graph as Alcohol % increases, Viscosity decreases continuously. It has much impact on viscosity. It is inversely proportional to viscosity.

So, it is conclude that catalyst concentration has maximum impact on viscosity. The viscosity of Mustard oil methyl ester was found to be within the ASTM limits. From table it is found that 0.5% catalyst by weight of Mustard oil, reaction time 40 minutes, reaction temperature 60˚C & alcohol 20% by weight of Mustard oil gives maximum yield and minimum viscosity shown in table. So, performance will be based on the Mustard Bio diesel obtained from the reaction of 0.5% catalyst by weight of Mustard oil, reaction time 40 minutes, reaction temperature 60˚C & alcohol 20% by weight of Mustard oil.

VI. CONCLUSION

The overall studies based on the production, fuel characterization of Mustard oil methyl esters were carried out. The following conclusions can be drawn:

1. The recovery of Mustard oil methyl ester of lowest kinematic viscosity (4.39 mm^2/sec) with 99.7% recovery is possible at reaction of 0.5% catalyst by weight of Mustard oil, reaction time 40 minutes, reaction temperature 60˚C & alcohol 20% by weight of Mustard oil.
2. The FFA content of Mustard oil methyl ester was found as 0.04% through titration process.

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