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Effect of Concentration of KOH, H_2O, Temp in In-situ Transestrification Reaction of Sesbania sesban, Capparis deciduas seed

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Abstract

Fatty acid methyl ester is defined as the mono-alkyl esters of fatty acids derived from vegetable oils or animal fats. In simple terms, it is renewable source of energy. Petrodiesel can be replaced by biodiesel due to its superiority. Biodiesel has more lubricity which increases lifetime of engine. It is less toxic and gives less emission of Carbon dioxide, Carbon monoxide, hydrocarbons and particulate matter. The biodiesel fraction from oil content of *Sesbania sesban, Capparis deciduas* are found 29% & 63.75 respectively. The percentage of biodiesel yield increases with concentration of KOH as a catalyst. The aim of this article is to demonstrate the cost effective new source of energy by single step reaction i.e. production of oil by combining extraction and reaction of extract with the mixture of alcohols. In this article the effect of catalyst concentration, time, water content and temperature on in-situ transestrification is studied to obtain optimum yield of fatty acid methyl ester (Biodiesel).Fuel characterization tests show the striking similarity of various physical & chemical properties and campers to ASTM standards.

Key words

Biodiesel; KOH; in-situ transestrification; Sesbania sesban; Capparis deciduas

1. Introduction

Biodiesel; a most important need of future can be obtained by in-situ transestrification reaction. The triglycerides are easily converted to monoalkyl esters of long chain fatty acids by this technique.
The reaction of reactive extraction is carried out by following three stages
(a) Conversion of triglycerides to diglycerides
(b) Conversion of diglycerides to monoglycerides
(c) Conversion of monoglycerides to biodiesel ester.

In in-situ transestrification seed powder is extracted with alcohol where alcohol acts as a solvent as well as reactant. This process reduces the cost of final product as this process has less number of unit operations. It is the best non-renewable source of energy with good environmental impact and easy recovery.

2. Material and Method

The seeds of *Sesbania sesban, Capparis deciduas* collected, cleaned and dried. The seeds are then grinded to fine powder by using heavy duty electric mixer of high rpm. Ten grams of seed powder was used as a starting material. It was mixed with mixture of methanol and ethanol. The in-situ transestrification with continuous stirring was carried out by adjusting 300 rpm oscillations. The heat is given by hot plate by keeping at 80^0 C for about 45 minutes. The solid cake and mother liquor were separated by vacuum filtration. A rotary evaporator was used for separation of solvent. The oil fraction separates at 80^0C. The oil content was preserved in airtight containers and used for further analysis.

The moisture content of dry seed powder and oil extracted by reactive extraction was obtained by Karl Fischer Titrator, µaquacal$_{100}$, manufactured by Analab Scientific Instruments Pvt. Ltd. During in-situ transestrification various concentrations of potassium hydroxide were used as a catalyst along with the mixture of alcohols. The reaction time was finalized for optimum yield is 45 minutes. The reaction was carried out at different temperatures. The temperature 80^0C gives maximum yield where as the oil was also separated at 80^0C by rotary evaporator. The water quantity also affects the rate of reaction considerably. Increase of aqueous medium reduces the yield of reactive extraction. The observed yield is maximum without addition of water. The separation of various components was studied by thin layer Chromatography. The best solvent for

separation was found to be Acetic Acid, Petroleum Ether and Ethanol with volume ratio 0.75: 7.25: 2.00. The silica gel suspended in chloroform and a pinch of plaster of paris was used for preparation of chromo plate. The spots were observed on chromatogram by keeping dry developed plate in iodine chamber. The areas of spots were calculated by usual method. These observations were used for calculation of percentage yield.

3. Results and Discussion

The percentage of biodiesel from *Sesbania sesban*, *Capparis deciduas* seeds were found to be 29%.percent (in oil fraction). The moisture content of oil obtained by Karl Fischer method was 0.56 percent and the moisture content of seed powder is around 0.4 percent. The optimum temperature for in-situ transestrification is 80^0C. The agitation was achieved by keeping 300 rpm oscillations continuously for 45 minutes optimized time.

The reactive extraction was studied for different concentrations of KOH, temperature and time intervals so as to obtain optimum conditions.TLC study of oil fraction obtained without heating and constant stirring did not show any spots. The chromatographic plate shows the positions of spots. All plates shows different number of spots. Some plates show only two spots.

The graph of effect of concentration of KOH clearly shows that the yield of biodiesel changes with change in the concentration of catalyst. The optimum yield of biodiesel was found at 0.1, 0.08 N Concentration of KOH.

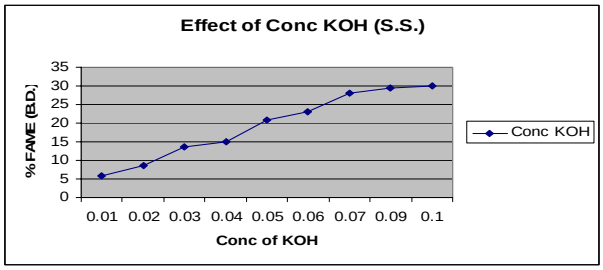

Figure 1. Effect of Conc KOH (S.S.)

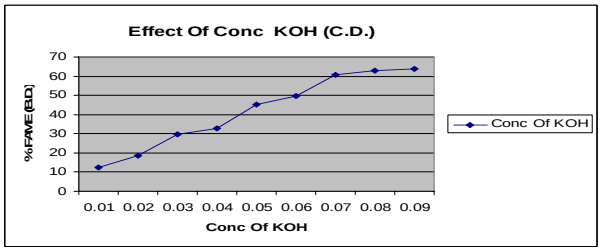

Figure 2. Effect of Conc KOH (C.D.)

The graph of effect of water quantity on reactive extraction clearly shows that the yield of biodiesel changes with change in the concentration of water. The optimum yield of biodiesel was found without water.

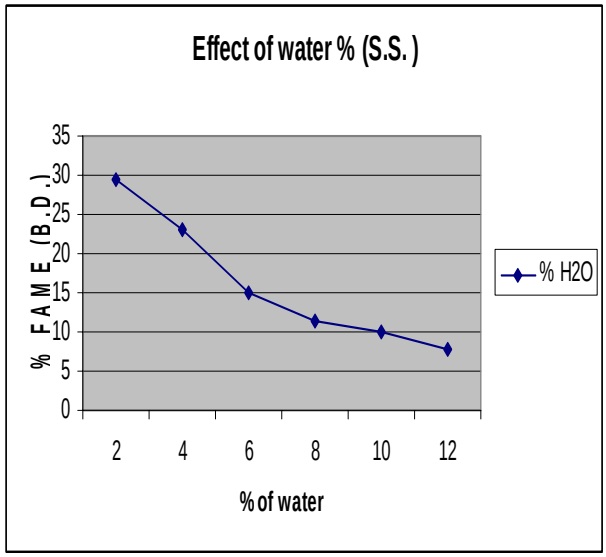

Figure 3. Effect of water % (S.S.)

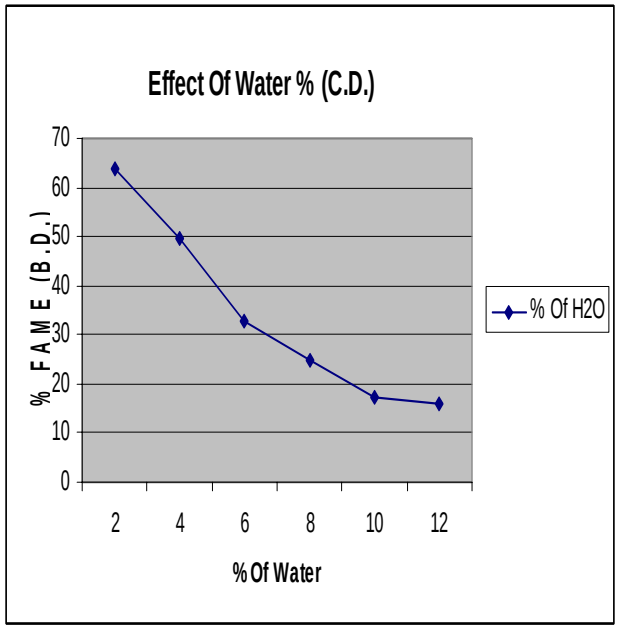

Figure 4. Effect of water % (C.D.)

The graph of effect of temperature on in-situ transestrification clearly shows that the yield of biodiesel changes with change in the temperature of the reaction mixture. The optimum yield of biodiesel was found at 80^0C temperature, yield decreases at higher as well as lower temperatures.

TABLE 1. Striking similarity of various physical & chemical properties and campers to ASTM standards *Sesbania sesban*

No	Performance Characteristics	Std .(FAME) B.D. ASTM	Sesbania sesban
1	Acid numbers, KOH/gm	0.80	0.26
2	Sulphated ash %	0.05% by	0.007
3	Sulfur, wt %,max	0.05 %	Nil
4	Phosphorus, wt%,	0.001 %	0.0059
5	Free/ Total glycerin.	0.020 %	Nil
6	Cloud point ASTM D2500	< 210	+6
7	Kinematic Viscosity,cSt@40c mm2/S	1.9-6.0 mm^2/S	0.5
8	Copper strip corrosion rating, max	No.3 max.	<3
9	Cetane Number	47	69.73

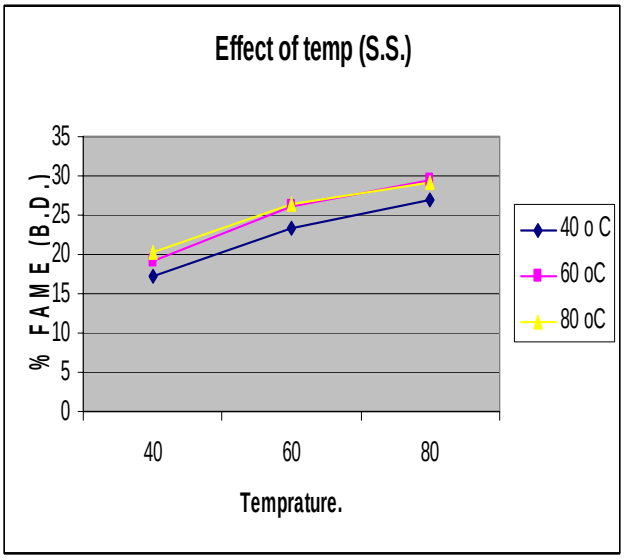

Figure 5. Effect of temp (S.S.)

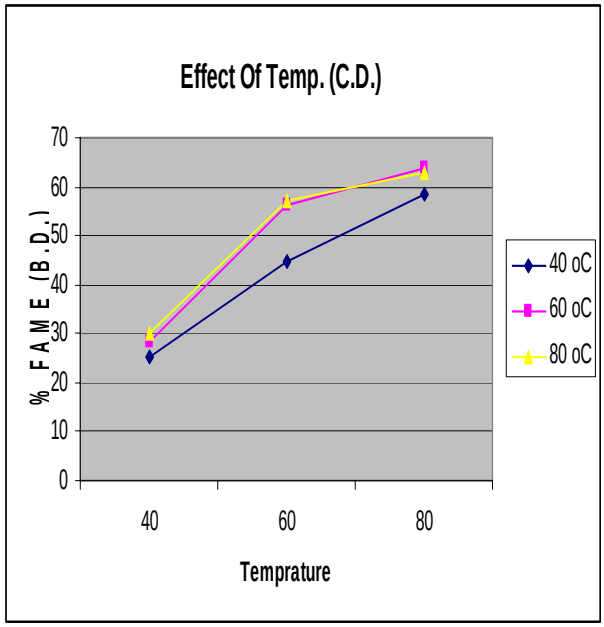

Figure 6. Effect of Temp. (C.D.)

Fatty acid methyl ester (Biodiesel) Fuel characterization tests (table 1) show the striking similarity of various physical & chemical properties and campers to ASTM standards *Sesbania sesban*

Fatty acid methyl ester (Biodiesel) Fuel characterization tests (table 2) show the striking similarity of various physical & chemical properties and campers to ASTM standards *Capparis deciduas*

TABLE 2. Striking similarity of various physical & chemical properties and campers to ASTM standards *Capparis deciduas*

No	Performanc Characteristics	Std .(FAME) B.D. ASTM	Capparis deciduas
1	Acid numbers, KOH/gm	0.80	0.43
2	Sulphated ash %	0.05% by	0.023
3	Sulfur, wt %,max	0.05 %	0.005
4	Phosphorus, wt%,	0.001 %	0.002
5	Free/ Total glycerin.	0.020 %	Nil
6	Cloud point ASTM D2500	< 210	+10
7	Kinematic Viscosity,cSt@40c mm2/S	1.9-6.0 mm^2/S	1.01
8	Copper strip corrosion rating, max	No.3 max.	<3

The fatty acid methyl ester (Biodiesel) of the *Sesbania sesban, Capparis deciduas* are tested for Acid numbers, , Sulphated ash %,Sulfur, wt %,max, Phosphorus, wt%, Free/ Total glycerin, Cloud point ASTM D2500,Kinematic Viscosity,cSt@40c,Copper strip corrosion rating,max, corrosion, Cetane Number. These results are tabulated and compared with ASTM standard the conclusions are drawn on the basis of standard. This plant can be used for the preparation of Fatty acid methyl ester (Biodiesel) which will be suitable for use replacement of diesel without any change in engine.

4. Conclusions

Biodiesel can be produced from the seeds of *Sesbania sesban, Capparis deciduas* by in-situ transestrification. The optimum concentration of KOH is 0.1 N, 0.08 N The biodiesel fraction from oil content was found 29%, 63.75 %. at 80^0C and 300 rpm oscillations for 45 minutes time and normal atmospheric pressure without addition of water in the reaction mixture. The harmful organic reagent like n-hexane was not at all used in this method.

This will be suitable for use replacement of diesel without any change in engine.

Hence this technique is environment friendly. The Biodiesel obtained has low cost and low viscosity. One can support the replacement of Petrodiesel by biodiesel; as it is easily recovered. The catalyst used is KOH which is cheap and easily available. Fatty acid methyl ester (Biodiesel) fuel characterization results are tabulated and compared with ASTM standard; the conclusions are drawn on the basis of standard. These plants can be used for the preparation of Fatty acid methyl ester (Biodiesel) which will be suitable for use as replacement of diesel without any modification in engine.

Acknowledgement

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