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STUDIES ON TRANSESTERIFICATION OF MAHUA (MADHUCA INDICA) SEEDS OIL

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***Abstract*— Animal fat, raw, and used vegetable oils have been explored to make bio-diesel (mono alkyl esters of long chain fatty acid) in order to substitute the dwindling supplier of conventional petro-diesel fuels. In the present investigation custard apple (Annoma Squamosha), seed oil (non-edible) was Transesterified with methanol in the presence of sodium hydroxide as catalyst. The transesterification reaction was carried out at 650C for an hour, keeping the molar ratio of methanol to oil at 6:1 and sodium hydroxide concentration of 0.5 wt % of the oil. The yield of fatty acid methyl esters produced under operating conditions was 86.4 wt%. The methyl ester produced by this reaction was analyzed to ascertain suitability as bio diesel fuels.**

***Index Terms*— Custard apple seeds oil, methanolysis, catalyst, temperature, condensation transesterification and bio diesel.**

I. INTRODUCTION

All developed countries include India are making efforts to search for suitable alternative diesel fuels that are environmental friendly. The need to search for these fuels arises mainly from the standpoint of preserving the global clean environment and the concern about long-term supplies of hydrocarbon based petro-diesel fuels. Edible vegetable oil [1,2] such as groundnut oil, sunflower oil, soya bean oil, palm oil, rapeseed and non-edible vegetable oil like Karanjia oil and Jatropa oil have been suggested as promising candidate for alternative diesel fuels. But their high viscosities, low volatilities and poor cold flow properties prevent them to be used as an alternative diesel fuel. The fatty acid methyl esters commonly known as bio diesel [3] have been suggested as alternative diesel fuels. Compared to edible vegetable oil for utilization of non-edible vegetable oil in the manufacture of bio-diesel seems to be economically feasible.

Authors [4-7] studied the process of transesterification of vegetable oils. They found that for the basic catalyzed transesterification reaction methanol/oil molar ratio of 6:1 was optimal in the temperature range 50-80°C.This results in a conversion of 70-90% (wt) fatty acid methyl ester, when 0.5-1% (wt) sodium hydroxide is used as catalyst.

The kinetics of transesterification of soya bean oil with 1-butanol and ethanol has been worked out by Freedman et al.[8].

Authors [9] reported the effect of temperature and variation in mixing intensity on the rate of transesterification of soyabean oil with ethanol. They have found that temperature range 60-80°C is suitable for transesterification process.

Krisnangkura et al.[10], transesterification palm oil with methanol using sodium methoxide as catalyst in the presence of Toluene. They observed that the conversion of palm oil increased with increase in molar ratio of methanol and palm oil in the range of 5.8 to 17.1.

Fig.1 Mahua flower

Authors [11] have reported the performance of conventional diesel engine operated on bio-diesel. They observed radiation in hydrocarbon and carbon monoxide emission in comparison to conventional diesel fuels. They have also reported lower particulate emission but polyaromatic hydrocarbons were slightly higher. The problem of crystallization of methyl esters at low temperature can be eliminated by winterization process.

Previous work done by the authors [12] has concerned on studies on transesterification of custard apple (Annona Squamosha) seeds oil. They have reported yields of 86.4% (wt) methyl ester at operating condition of temperature is equal to 65 0C, molar ratio of methanol to oil 6:1 and sodium hydroxide concentration of 0.5% (wt) of the oil.

The objective of this work is to make bio - diesel using Mahua [13-15] seeds oil (non-edible) by transesterification process. Mahua is a medium to large size tree of family Sapotaceae with a wider canopy. The tree may attain height of up to 20 meters. It is found in mixed deciduous forest usually of somewhat dry type, often growing on rocky and sandy soil and thriving on Deccan trap. It is common throughout the central India, Maharashtra and Andhra Pradesh. It is also planted in many parts of India. Among the non-edible oil seeds, crops. A sample of Mahua flower in shown in Fig.1.Mahua have gained relatively more interest in view of the fact that the seed Kernel contain 50% (wt) oil, which is higher compared to other non-edible sources. A sample of Mahua seed is shown in Fig.2.

The further objective of this work is the verification of the properties of the bio-diesel obtained experimentally and is compared with the Indian requirement [16] for high-speed diesel fuels.

Fig.2 Mahua seeds

II. EXPERIMENTAL

Transesterification reaction has been carried out using Mahua seeds oil and methanol in the presence of sodium hydroxide as catalyst.

Experimental Setup: The experimental apparatus used to carry out transesterification reaction consists of a cell that contains two necked flat bottom flask, reactants, thermometer pocket, thermometer, vertical condenser and magnetic stirrer cum heater. The complete experimental setup is shown in Fig.3.

Experimental Procedure

The required amounts of Mahua seeds oil was placed in the apparatus to perform the transesterification reaction at operating temperature by regulating the power supply through magnetic stirrer cum heater. A known amount of sodium hydroxide was dissolved in a fixed quantity of methanol and this solution was added to the oil. These amounts of oil, methanol and the catalyst ensure the molar ratio of methanol to oil to be 6:1 and sodium hydroxide to be 0.5% (wt) of the oil. The magnetic stirrer was turned on at a certain RPM and the reaction mixture was stirred vigorously as oils and methanol were immiscible. The reaction was carried on for 1 hour with continuous stirring. The reaction mixture was then transferred into a separating funnel and was allowed to cool overnight. Two layers were formed. The bottom layer consisted of Glycerol and Methanol and the upper layer had methyl ester with little amount of methanol. The lower layer was drained off and the upper layer was treated with Sulphuric acid 20% (wt) to deactivate the catalyst. It was then washed five times, each time with 10 ml distilled water. Washing was done to remove the catalyst. The washed layer was dried with anhydrous calcium chloride and filtered. The filtrate was distillated under vacuum to recover methanol. The lower glycerol layer was also distilled under vacuum to recover methanol.

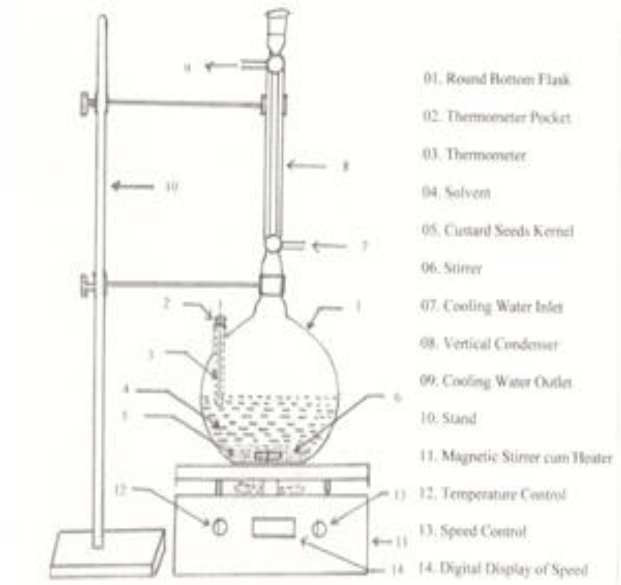

Fig.3 Schematic diagram of Experimental setup

III. RESULTS AND DISCUSSION

A. Composition of Mahua Seeds Oil

The fatty acid compositions of Mahua seeds oil were analyzed by Gas chromatograph. The result of the analysis is shown in GC Chromatogram Fig.4. Since the sample is separated in the column, the different peaks indicated by retention time on the chromatogram corresponds to different components of the oil sample. For example, the retention time on the peak 22.72 is Oleic acid. The area under the peaks indicates the percentage composition of individual components and the calculated percentage composition is given in Table 1. This table shows that the major Fatty acids in custard seeds oil are Palmitic acid, Stearic acid, Oleic acid, Linoleic acid and Linolenic acid. In addition to these acids, traces of Caproic acid, Caprylic acid, Capric acid, Lauric acid and Myristic acid are also present. It is seen from the table that among the major Fatty acid Oleic acid is maximum (48.08% (wt)) and that of the Myristic acid is minimum 0.25% (wt). On the other hand, among the traces of Fatty acids maximum is Caprylic acid 0.05% (wt) and that of minimum is Capric acid 0.02% (wt).

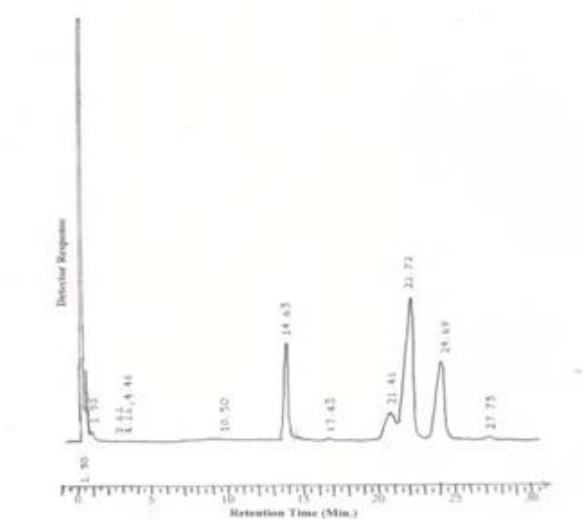

Fig.4 GC Chromatogram of Mahua seeds oil

Table 1: Composition of Mahua Seeds Oil

Fatty Acids	Weight Percentage
Caproic Acid	00.05
Caprylic Acid	00.05
Capric Acid	00.02
Lauric Acid	00.00
Myristic Acid	00.25
Palmitic Acid	15.87
Stearic Acid	12.58
Oleic Acid	48.08
Linoleic Acid	22.77
Linolenic Acid	00.56

B. Yield Of Fatty Acid Methyl Ester

The amount of reactants and catalyst required for the transesterification reaction and product formed are given in Table 2. This table indicates that the mass balance is satisfied for the transesterification reactions conducted for Mahua seeds oil. The yield of Fatty acid Methyl ester was found to be 86.4% (wt), which is little less compared to findings of other investigators. The low yield of Mahua seeds oil methyl ester was mainly due to inefficient mixing of the contents during the course of reaction.

Table 2: Mass Balance of Transesterification reaction

Reactants	Quantity (g)
Custard Seeds Oil	905.20
Methanol	616.30
Sodium Hydroxide	4.52
Products	Quantity(g)
Methyl ester	876.23
Glycerol	206.38
Methanol Recover	428.45
Percentage error	9.8×10^{-1}
Yield of Methyl ester {wt%)	96.80

C. Properties Of Mahua Seeds Oil And Their Methyl Esters

Various fuel properties of Mahua Seeds Oil and their Methyl Esters were determined experimentally to ascertain their suitability as Diesel fuels. These properties are given in Table 3. A sample of Bio-diesel made from Mahua seeds Kernel oil is shown in Fig.5.

Table 3: Characteristics of Mahua Seeds Oils and Methyl Ester

Characteristics	Custard seed oil	Methyl ester	BIS Requirement of Diesel fuels	Method of test
Density @ 25° C (kg/m^3)	905.2	852.6	------	IS:1448 (P:16)
Acid Value (mg KOH/g)	0.322	0.132	0.5	IS:548-1964 (Part-1)
Free Fatty Acids % (wt)	1.90	0.042	------	IS:548-1964 (Part-1)
Kinematics Viscosity @ 25°C (C St.)	37.18	20.1	2--7.5	IS: 1448-1976(P:25)
Water Content (Vol. %)	NIL	NIL	0.25	IS:1448-1967(P:40)
Flash Point (°C)	241	152	66	IS:1448 (P:20)
Pour Point (°C)	4	-3	-6	IS:1448-1970(P:10)
Cloud Point (°C)	6	-1	-5	IS:1448-1970(P:10)
Distillation Range,90% (°C)	------	------	366	IS:1448-1967(P:18)
Cetane Index	50	45	42	------
Energy Content (MJ/Kg)	38.86	41.03	------	------
Iodine Value	120	85	------	IS:548-1964 (Part-1)
Saponification value	196	186	------	IS:548-1964 (Part-1)
Carbon Residue (wt %)	3.652	1.62	1.5	IS:1448 (P:8)
Ash (wt %)	0.021	0.01	0.02	IS:1448 (P:4)
Referective Index	1.452	-------	------	------

Fig.5 Mahua Seeds Bio-Diesel

IV. CONCLUSIONS

This study suggest that the Mahua seeds oil can be used as a source of triglycerides in the manufacture of bio-diesel by the process of transesterification. The use of Mahua seeds oil seems to be very effective in regards to global environmental maintenance and resource cycling considerations. The bio-diesel from the Mahua seeds oil meets the Indian requirements of high-speed diesel oil. Bio-diesel derived from the oil which is renewable, bio-degradable, non-toxic, essentially free from Sulphur and Aromatic compound and safe to handle.

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