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STUDIES ON TRANSESTERIFICATION OF CRUSTARD APPLE (ANNONA SQUAMOSHA) SEEDS OIL

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***Abstract*—Animal fat and 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 petrodiesel fuels. In the present investigation custard apple (Annona Squamosha) seeds oil (nonedible) was transesterified with Methanol in the presence of sodium hydroxide as catalyst. The transesterification reaction was carried out at 650C for one hour, keeping the molar ratio of methanol to oil at 6:1 and sodium hydroxide concentration of 0.5% by wt of the oil. The yield of fatty acid methyl esters produced under operating conditions was 87.3% by wt. The Methyl ester produced by this reaction was analyzed to ascertain suitability as Bio-diesel fuels.**

***Index Terms*— Custard apple seed oil, Methanol Catalyst, Temperature, Condensation, Transesterification and Bio-diesel.**

I. INTRODUCTION

All developed countries including India are making efforts to search for suitable alternative diesel fuels that are environment friendly. The need to search for these fuels arises mainly from the stand point of preserving the global clean environment and the concern about long term supplies of hydro carbon based petrol-diesel fuels. Vegetable oils, edible and non-edible [1,2] such as ground nut oil, sunflower oil, palm oil, rape seed oil, Karanjia oil and Jatropa oil have long been suggested as promising 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 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 50-78^{0}C. This results in the conversion of 70-95% by wt, when 0.5 -1% by wt sodium hydroxide is used as catalyst.

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

Noureddin and Zhu [9] reported the effect of temperature and variation in mixing intensity on the rate of transesterification process. Krishnangukura et.al [10], Transesterified 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 5.8-17:1

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

The objective of this work is to make bio-diesel employing custard Apple seeds (nonedible) oil by transesterification process. Custard apple (Annona Squamosha) commonly known as Sitaphal [12] is one of the finest fruits introduced in India from tropical America. A cut section of custard apple fruit is shown in Fig.1

Fig 1.Cut section of custard apple fruit

It is cultivated in Andhra Pradesh, west Bengal and many other parts of India. It is also found in wild state. Among the oil seed crops custard Apple seeds have gained relatively less

interest in view of the fact that it is unfamiliar and seeds kernel contain 40% oil by wt [13,14] which is less compared to other edible sources.

The further objective of this work is the verification of properties of the bio-diesel obtained experimentally and to be compared with the Indian requirement [15] for high speed diesel fuels.

II. Materials and Methods

A. Material/Instruments Used

The materials/instruments used for this work are flat bottom flask, basket heater, distillation unit, thermometer, measuring cylinder, conical flask, separating funnel, gas chromatograph.

B. Sample Collection

The custard apple seeds are collected from local market, Bangalore. A sample of custard apple seed is shown in Fig.2

Fig.2. Custard apple seeds

C. Extraction of Oil from custard apple seeds

The custard apple seeds are dried under sunlight for two to three days to remove the excess moisture present in it. After that the kernels are separated and grinded using a mixer grinder to a particular particle size. Then, oil is extracted by Soxhlet apparatus. GC analysis of custard apple seed oil is shown in Fig.3.

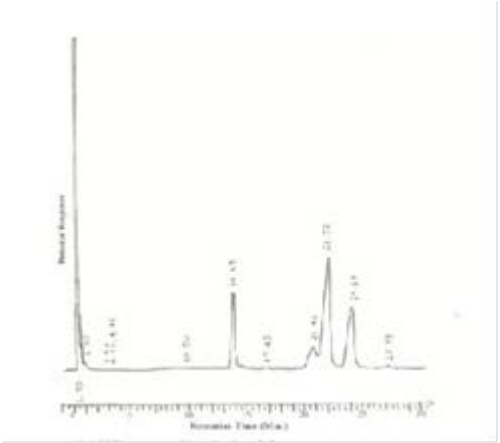

Fig.3. GC Chromatogram of Custard Seed oil

Since the sample is separated in the column, the different peaks indicated by retention time on the chromatogram correspond to different component of the oil sample.

Table2. Composition of fatty acid in custard seed oil

Fatty acids	Weight Percentage
Caproic acid	0.04
Caprillic acid	0.05
Capric acid	0.02
Lauric acid	0.03
Myristic acid	0.25
Palmitic acid	16.25
Stearic acid	10.31
Oleic acid	47.91
Linoleic acid	24.31
Linolenic acid	0.83

For example, the retention time on the peak 22.72 is oleic acid.The area under the peaks indicates the percentage composition of the individual component and the calculated percentage composition is given in Table2.The table shows that the major fatty acids in custard seeds oil are palmatic acid, strearic acid, Oleic acid, Linoleum acid and Linolenic acid. In addition to these acid traces of Caproic acid, Caprillic acid, Capricorn 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 47.91% by wt and that of the Myristic acid is minimum 0.25% by wt. On the other hand, among the traces of fatty acid maximum is Caprillic acid 0.05% by wt and that of the minimum is Capric acid 0.02% by wt.

D. Experimental setup for Transesterification

The experimental setup 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 experimental setup is shown in Fig.4.

E. Experimental Procedure

The required amounts of custard seeds oil is placed in the apparatus to perform the transesterification reaction at operating temperature by regulating the power supply through magnetic stirrer cum heater. 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 6:1and sodium hydroxide to be 0.5% by wt of the oil. The magnetic stirrer is turned on at a certain RPM and the mixed solution is stirred vigorously for one hour. The reaction mixture was then transferred in a separating funnel and is allowed to cool overnight. Two layers are formed. The bottom layer consisted of glycerol and methanol and the upper layer had methyl Ester with little amount of methanol. The lower layer is drained off and the upper layer is treated with sulphuric acid 20% by wt to deactivate the catalyst.It is then washed five times, each time with 10ml of distilled water. Washing is done to remove the catalyst. The washed layer is dried with anhydrous calcium chloride and filtered. The filtrate is distilled under vacuum to recover methanol. The lower glycerol layer is

also distilled under vacuum to recover methanol. The yield of fatty acid methyl Ester was found to be 86.4wt%, which is little less compared to findings of other investigators. The low yield of custard Apple seeds oil methyl ester was mainly due to inefficient mixing of the contents during the course of reaction.

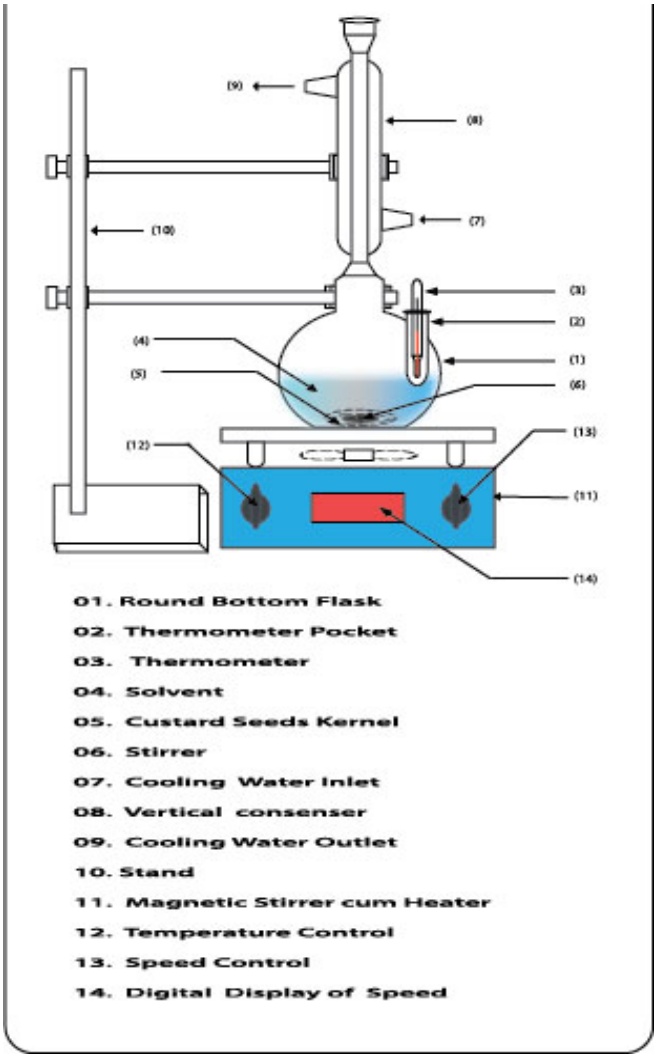

Fig 3. Schematic Diagram of experimental setup

III. Results and Discussions

A. Yield of Fatty Acid Methyl Ester

The amount of reactant and catalyst required for the Transesterification reaction and product formed are given in Table2. This table indicates that the mass balance is satisfied

Table2:Mass balance of Transesterification process

Reactants	Quantity (g)
Custard Seeds Oil	908.80
Methanol	198.07
Sodium Hydroxide	4.54
Products	
Methyl ester	785.20
Glycerol	220.06
Methanol Recover	95.48
Percentage error	0.96
Yield of Methyl Ester (wt%)	86.39

for the Transesterification reaction conducted for custard apple seed oil. The yield of fatty acid methyl ester was found to be 87.3% by wt.which is little more compared to the finding of other investigators. The high yield of custard apple seed oil methyl ester was mainly due to sufficient mixing of the contents during the course of reaction.

B. Properties of Custard Apple Seeds Oil and their Methyl Esters:

Table3.Characteristics of custard apple seed oil methyl ester

Characteristics	Custard seed oil	Methyl ester	BIS Requirement of Diesel fuels	Method of test
Density @ 25° C (kg/m^3)	905.3	852.7	------	IS:1448 (P:16)
Acid Value (mg KOH/g)	0.321	0.133	0.5	IS:548-1964 (Part-1)
Free Fatty Acids % (wt)	1.91	0.041	------	IS:548-1964 (Part-1)
Kinematics Viscosity @ 25°C (C St.)	37.17	20.11	2--7.5	IS: 1448-1976(P:25)
Water Content (Vol. %)	NIL	NIL	0.25	IS:1448-1967(P:40)
Flash Point (°C)	242	153	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	44	42	------
Energy Content (MJ/Kg)	38.87	41.05	------	------
Iodine Value	121	85	------	IS:548-1964 (Part-1)
Saponification value	197	185	------	IS:548-1964 (Part-1)
Carbon Residue (wt %)	3.653	1.63	1.5	IS:1448 (P:8)
Ash (wt %)	0.021	0.01	0.02	IS:1448 (P:4)
Refractive Index	1.452	-------	------	------

Various fuel properties of custard apple 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 produced during investigation is shown in Fig.5

Fig.5 Sample of custard apple seed bio-diesel

IV. Conclusions

This study suggests that the custard Apple seeds oil can be used as a source of triglycerides in the manufacture of bio-diesel by the process of transesterification. This use of custard apple seeds oil seems to be very attractive in regards to global environmental maintenance and resource cycling considerations. The bio-diesel from the custard apple seeds oil meets the Indian requirements high speed diesel oil. Bio-diesel derived from this oil is renewable, biodegradable, non -toxic, essentially free from sulphur and aromatic compound and safe to handle.

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