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"Biodiesel Production From Custard Apple Seed (Annona Squamosa) Oil And Its Characteristics Study"

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Abstract

Now a day's increasing in prices and depletion of fossil fuels, creates very necessary to find out an alternative fuel (biodiesel) from non-edible oil seeds. This paper deals with the transesterification of custard apple seed oil by means of methanol in presence of Potassium hydroxide catalyst at less than 65˚C.The viscosity of biodiesel produced from custard apple seed oil is nearer to that of the commercially available diesel. The custard apple seed oil is characterized by GC (gas chromatography) analysis and the important properties of biodiesel such as density, flash point, cloud point, pour point and kinematic viscosity, ash content, carbon residue are found out and compared with that of ASTM-biodiesel standards and commercially available diesel. The study encourages the production of biodiesel from Custard Apple seed (Annona squamosa) Oil and value addition of custard apple fruit.

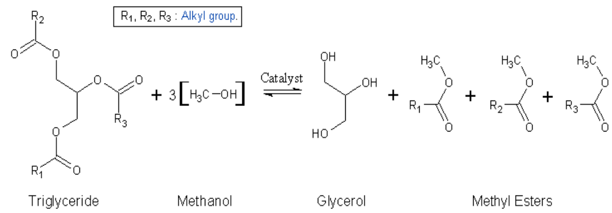
Key words: biodiesel, custard apple seed oil, transesterification.

"1. Introduction":

Energy consumption is constantly increasing all over the world in spite of the rationalization measures that have been undertaken [1]. Liquid fossil fuels are the main and most frequently used fuels for mobile machinery. Considering the fact that the entire development of mobile machinery is based on the use of liquid fossil fuel, it is difficult to expect a shift from this trend to a mass development and use of new engine constructions that would be suitable for some other type of fuel. The studies have been focused on discovering the fuel that would be adaptable to the existing engine constructions and that would meet the criteria regarding renewability, ecology and reliability of use. Fulfillment of the mentioned criteria is the basis for a successful fossil fuel replacement by some other types of fuel. During the last decade biodiesel has become the most common renewable liquid fuel due to its possibility to meet the set requirements of the previously mentioned criteria. Namely, the use of biodiesel does not require any type of engine modifications or modifications of the fuel injection system. The exceptions are older engine constructions which need a replacement of sealant and fuel injection hose [2].

Custard apple as shown in figure-1 belongs to the family *Annonaceae* with botanical name *Annona squamosa*, is commonly found in deciduous forests, also cultivated in wild in various parts of India. It is a native of West Indies; now cultivated throughout India and other tropical countries. Literatures of many research works prove that every parts of Annona *squamosa* possesses medicinal property [3-6]. It had also been known as sweetsop and sugar apple (English), seetaaphala and amritaphala (Kannada), Atoa and shariffa (Hindi) [7]. *Biophysical limits*:-Altitude 0-2000 m, Mean annual temperature Up to 41˚C, Mean annual rainfall Above 700 mm, Soil type Annona squamosa tolerates a wide variety of soils, it grows in rich, well-drained, deep rocky soils but prefers loose, sandy loams [8]. It has been reported that Annona squamosa seeds as figure-2 contains 26.8% [9].Residual agricultural products and food-processing by-products or wastes are often considered a problem. After processing, a large amount of plant material often remains without any application. The conversion of such materials into valuable resources can be a good contribution to a reduction in residues. In this sense, residue valorization has become of great interest from an economical point of view [10].The transesterification reaction formally requires a molar ratio of alcohol to oil of 3:1 but in practice a molar ratio of 6:1 needs to be applied for the reaction to proceed properly to high yield. The transesterification usually requires about 1hour at normal pressure with the reaction temperature 60-65˚C (for methanol) [11].

1.1. Biodiesel Transesterification Reactions: Transesterification process consists of a sequence of three consecutive reversible reactions i.e. conversion of triglycerides to diglycerides followed by diglycerides to monoglycerides. The glycerides were converted into glycerol and one ester molecule at each step. The methanolysis transesterification reaction is represented in equation below. [12].



If the oil contains more than 4% free fatty acids (FFA), then a two step transesterification is applicable to convert the high FFA oils to its mono esters [13]. The first step, the acid catalyzed esterification reduces the free fatty acid content of the oil. The second step, alkaline transesterification process converts the products of the first step to its mono-esters and glycerol[14].

"2. Experimental Methods":

2.1. Extraction of oil: The extraction of oil from custard apple seeds was done by using mechanical expeller and solvent extraction by soxhlet apparatus using methanol as solvent. The yields were given in table-1.

2.1.1. Soxhlet extraction(Laboratory scale): To find out oil content of custard apple seeds

Steps:-1 .50grams of fully ripened custard apple seeds crushed and taken in the testing setup.
2. 150ml of methanol taken in the round bottom flask as solvent.
3. Kept reaction at 65˚c for number of cycles.
4. Methanol recovered by retaining oil in round bottom flask.
5. Oil in the flask transferred to measuring beaker and measured.
6. The quantity of oil obtained is estimated to 1kg of seeds roughly.

Obtained results during experiment:

For 50gram seeds -13ml of oil obtained for 30 cycles. (For custard apple seeds, 1cycle time=5min36sec).1000gram of seeds=260ml oil. The percentage of oil content obtained is 26%.

2.1.2. Mechanical Expeller (production scale):

Steps:-1. Seeds are crushed using mechanical expeller to get oil. (Mechanical expeller is of capacity 30kg/hr, 3passes).
2. For a batch 5kg of seeds are taken to crush, each batch of seeds passed 5times to obtain complete oil.
3. Oil obtained was collected in a glass reagent container by filtering it with filters.
4. Filtered oil left for 10-12hrs for settling of minute dust particles.
5. After filter and settling, oil stored in a reagent glass bottles.

2.2. Determination of free fatty acid (FFA) content in raw oil:-

2.2.1: Preparation of 0.1Normal (0.1N) NaOH solution:

4 grams of NaOH is weighed & transferred into the conical flask containing 1 liter of water. Dissolve NaOH completely by constant stirring then the solution we get is called 0.1N NaOH solution. (With NaOH the molecular weight is 40 so a 0.1N solution contains 4g per liter)

2.2.2: Titration and calculation of free fatty acid content in raw oil:

Take 25ml of 0.1N NaOH solution in the burette and then take 10 grams of custard apple seed oil, in a conical flask, add 50ml of Isopropyl alcohol into the conical flask and also add 5-6 drops of Phenolphthalein as indicator and shake well. This is titrated against the 0.1N NaOH solution until it turns pink color, this is the indication of end point and by using the formula we can find FFA content in the oil.

2.2.3: FFA calculation:

$$\text{FFA Content} = \frac{28.2 \times Normality\ \ of\ NaOH \times Titrati\ on\ value}{weig\ ht\ of\ the\ oil}$$

$$\text{FFA Content} = \frac{28.2 \times 0.1 \times 6.3}{10.5\text{gm}} = 1.692$$

Note that the above formula contains 28.2 which is the molecular weight of oleic acid divided by ten. Oils are not made of only oleic acid hence this formula results in small errors, normally accepted.

2.3. Transesterification:

Procedure: Take 1ltr of custard apple seed oil in a three neck flask with reflux condenser, heat the oil up to 60°C add 300ml of methanol and 10gms of potassium hydroxide catalyst. Run the process for about 90minutes as shown in Figure-1.Transfer that oil into separating funnel, allow it to settle for about 7-8 hours then two layers will be formed as shown in figure-3 below, upper layer is biodiesel and lower layer is glycerin. Separate the glycerin and biodiesel. **The Yield after this process was found to be 88-90% of biodiesel. (850-900ml)**

2.4. Determination of physiochemical properties:

2.4.1. Viscosity: Kinematic viscosity is the resistance offered by one layer of fluid over another layer. The viscosity is important in determining optimum handling storage, and operational conditions. Fuel must have suitable flow characteristics to ensure that an adequate supply reaches injectors at different operating temperatures. High viscosity can cause fuel flow problems and lead to stall out.

Procedure:

Fill the bio-diesel in the cannon– fenske viscometer [tube no 100, direct type] bulb as shown in figure-5. Insert the viscometer tube in the viscometer-water-bath apparatus. Heat the oil to 40°c and maintain the temperature for a period of 20-30 min. After 30 min open the tube, suck the oil and simultaneously start the stopwatch when the oil reaches starting point mark. Stop the stopwatch once the oil flow reaches the bottom mark in the bulb. Note the seconds on the stopwatch.

Kinematic viscosity in Centistokes (Cst) =

$$(\text{Number of seconds} \times \text{standard factor of the bulb viscometer used for testing})$$

[Note: The standard viscometer factor as specified by manufacturer is 0.0238]

2.4.2 Density: A hydrometer is the instrument used to measure the specific gravity (relative density) of biodiesel. That is the ratio of the density of water. The hydrometer is made of glass and consists of a cylindrical stem and bulb weighed with mercury or lead shot to make it float upright. The hydrometer contains a paper scale inside the stem, so that the specific gravity can be read directly.

Procedure: Measure 500 ml of the Bio-Diesel in a clean & dry measuring cylinder. Allow the Bio-Diesel to settle. Gently lower the hydrometer in to the biodiesel in the cylinder until its floats freely. Note the point at which the surface of the Bio-diesel touches the stem of the hydrometer. Reading will be specific gravity multiplied by thousand will give density.

2.4.3. Flash point: The lowest temperature at which the vapor of a combustible liquid can be made to ignite momentarily in air is identified as the flash point and correlates to ignitibility of fuel. Low flash point can indicate residual methanol remaining from the conversion process. The flash point is often used as a descriptive characteristic of liquid fuel and it is also used to characterize the fire hazards of liquids. "Flash point" refers to both flammable liquids and combustible liquids.

Procedure: Pour measured biodiesel up to the mark indicated in the flash point apparatus, Heat the oil & stir the oil at regular intervals. Introduce external fire near the opening provided in the apparatus at regular period till a flash is observed. Once the flash is observed note the temperature. The noted temperature at the time of flash is the flash point of biodiesel. The setup used for finding out flash point is as shown in figure-6.

"3. Results and Discussions:"

The percentage composition of free fatty acids present in custard apple seed oil was obtained by gas chromatographic analysis and is represented in table-2. In gas chromatography result table we can seen that oleic acid (39.72%) is the major constituent.

The following physiochemical properties listed of biodiesel produced were determined and compared with ASTM biodiesel standard values and commercially available diesel and the results are represented in table-3.

3.1. Viscosity: Among the general parameters for biodiesel the viscosity controls the characteristics of the injection from the diesel. Viscosity value of custard apple biodiesel is in range of ASTM biodiesel standards.

3.2. Density: Higher densities of custard apple biodiesel as compared to commercially available diesel may be attributed to the higher molecular weights and triglyceride molecules present. Density of custard apple biodiesel lies in the range of ASTM biodiesel standards.

3.3. Flash Point: Flash point of custard apple biodiesel is nearer to ASTM biodiesel standards.

3.4. Calorific value: The calorific value is the energy released in the form of heat when an hydrocarbon (fuel) undergoes complete combustion with oxygen under standard conditions. The chemical reaction is typically a hydrocarbon reacting with oxygen to form carbon dioxide, water and heat. The obtained calorific value is in range of ASTM biodiesel standards.

3.5. Graphs: Below mentioned graphs 1, 2& 3 shows comparison of flashpoint, viscosity and density for custard apple seed oil, custard apple biodiesel and commercial diesel.

"4. Conclusions": By observing all results of biodiesel from custard apple seed oil, custard apple seeds can be used as biodiesel feed stock. All the properties are in the range of ASTM biodiesel standards, this can be promising factor to use custard apple seeds as one of the biodiesel source.

Table-1: comparison of oil extraction [between mechanical and chemical]

Quantity	Type of Extraction	
	Mechanical expeller	Soxhlet extraction
1kg of seeds	180-200ml of oil	250-260ml of oil

Table-2: The fatty acid composition of custard apple seed oil by gas chromatography

SI NO.	PARAMETERS	RESULTS
1	Caproic Acid	Nil
2	Caprillic Acid	Nil
3	Capric Acid	Nil
4	Lauric Acid	0.08%
5	Myristic Acid	Nil
6	Palmitic Acid	17.79%
7	Stearic Acid	4.29%
8	Oleic Acid	39.72%
9	Linoleic Acid	29.13%
10	Linolenic Acid	1.37%
11	Arachidonic Acid	1.06%
12	Behenic Acid	2.01%
13	Erucic Acid	Nil
14	Lignoceric Acid	Nil

Table-3: The physicochemical properties of custard apple biodiesel comparisons with the ASTM biodiesel standard and commercially available diesel.

SI No	PROPERTIES	UNITS	EXPERIMENTAL VALUES			
			BIODIESEL (custard apple)	BIODIESEL STANDARD VALUE(ASTM)	COMMERCIALLY AVAILABLE DIESEL	PROTOCALL
1	Kinematic viscosity@40˚c	centistokes	5.712	1.9-6.0	2.54	ASTM D445
2	Density	Kg/m^3	865	870-900	820	ASTM D4052
3	Flash point	˚C	150	130	54	ASTM D93
4	Calorofic value	KJ/Kg K	37510.8	37000 to 42500	43500	ASTM D240
5	Cloud point	˚C	2	-3 to 12	-28 to -7	ASTM D2500
6	Pour point	˚C	5	-15 to 10	5.6 to 11.1	ASTM D97
7	Ash content	%w/w	0.02	-----	0.02max	ASTM D482
8	Carbon Residue	%w/w	Nil	-----	0.05max	ASTM D524

Figure-1:Annona squmosa fruit

Figure-2:custard apple seeds

Figure-3: Transesterification setup

Figure-4: Separation of two layers

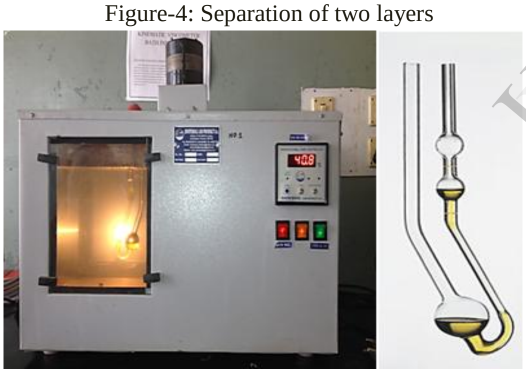
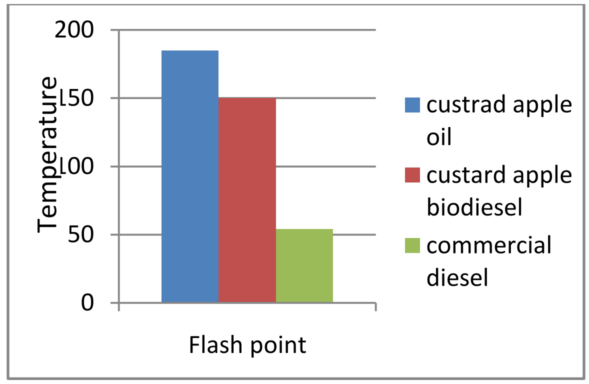
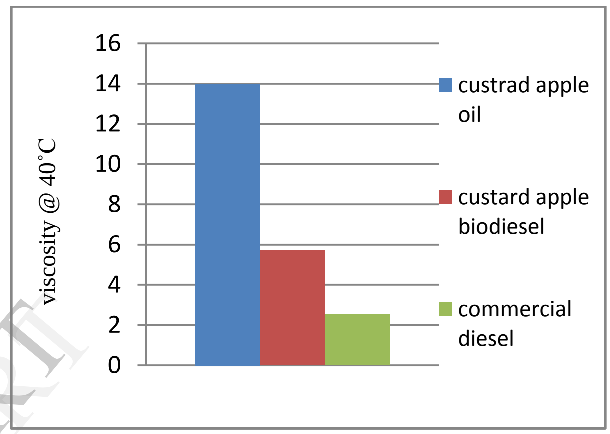


Figure-5: Kinematic viscosity bath
Instrument with cannon fenske tube

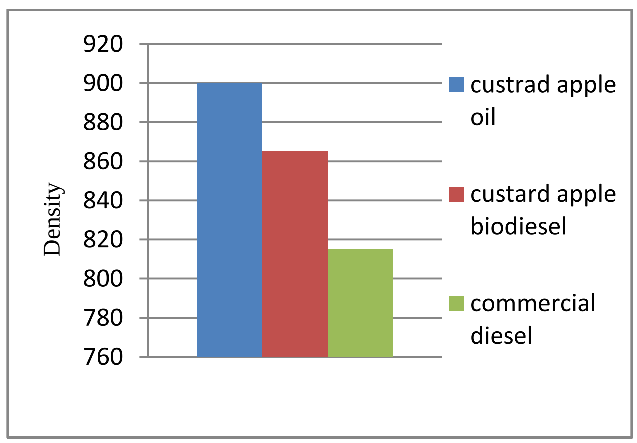
Figure-6: Pensky Martin Flash point instrument



Graph1: Flash point comparision



Graph2: Comparison of Viscosity @40˚C.



Graph3: Comparision of Density

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