Optimization of conditions for the extraction of Biodiesel from Jatropha curcas

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***Abstract*: Biodiesel is known as the green-fuel which is non-toxic and eco-friendly. The biodiesel is produced from Jatropha seeds by the process of transesterification. These seeds act as the main sources in the production of biodiesel. As in current scenario, the cost of all commercial fuels are of higher cost and the rates are increasing dramatically day by day and even their usage leads to pollution , that in turn increases the global warming. So, biodiesel usage would be the only remedial way that leads to the environmental friendly fuel and a cost-effective fuel. The existing method for the extraction of oil from seeds involves the solvent extraction method using different solvents. Using different combinations of solvent in order to determine the efficiency yield of oil from seeds. The yield percentage of the biodiesel is increased from the extraction method of oil. The best solvent and remedial measure of solvent was determined by the analysis. The estimation of biodiesel sample was carried out with Thin Layer Chromatography (TLC) and Gas Chromatography Mass Spectrometry (GC-MS) in order to determine presence of fatty acids in biodiesel. This method is an alternative for the extraction of oil from seed using solvent extraction.**

Keywords:Biodiesel,transesterification,Jatrophacurcas,Solventextraction

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I. INTRODUCTION

Biodiesels availability and low cost of petroleum diesel fuel, vegetable oil-based fuels gained little attention, except in times of high oil prices and shortages. World war 2 and the oil crises saw brief interest in using vegetable oils to fuel diesel engines, in 1970's. Due to higher viscosity of traditional vegetable oil, the newer diesel engine designs could not run on traditional vegetable oils, compared to petroleum diesel fuel. To solve this problem an innovative method was needed to lower the viscosity of vegetable oils to a point where they could be burned properly in the diesel engine. Many methods have been proposed to perform this task, including pyrolysis, blending with solvents, and emulsifying with water or alcohols, none of which have provided a suitable solution. In the year 1937 it was a Belgian inventor who first proposed using transesterification to convert vegetable oils into fatty acid alkyl esters and use them as a biodiesel.

II. BIODIESEL IN ENVIRONMENTAL USE

The biodiesel was developed by inventor Rudolph diesel in the year 1890s which has become a choice of power, reliability and high fuel economy over the world wide. In the early experimental studies the French government and Dr. Diesel envisioned that pure vegetables oils could power early diesel engines for agriculture in remote areas of the world, where petroleum was not available at that time. Modern biodiesel involves the conversion of vegetable oil into a compound called fatty acid methyl esters, research conducted in Belgium 1930's, but today's biodiesel industry was not established in Europe until the late 1980's.

The diesel was developed for a particular reason to improve efficiency, cumbersome and dangerous steam engines of the late 1800s. The diesel works on the principle of compression ignition, in which fuel is injected into the cylinder after air has been compressed to a high temperature and pressure. This mainly involves the conversion of chemical energy in the fuel into mechanical energy. Dr. Rudolph diesel holds the first patent for the compression ignition engine in 1893. Diesel became known to worldwide for his innovative engine which could use variety of fuels.

III. OIL EXTRACTION PROCESS

The seeds were collected, sun dried and made into crushed mixtures using the mortar and pestle. 50g of crushed seeds (*Jatropha curcas*) were taken and covered within the muslin cloth. The soxhlet apparatus was setup and the distillation column was filled with the solvents chosen for the extraction process. About 200ml of solvents such a Hexane, Isopropyl Alcohol and Petroleum Ether were used to fill the distillation flask. The muslin cloth with the Jatropha seeds mixtures was packed into the condenser column.

Once the apparatus was setup, switch ON the heating mantle by setting the temperature equivalent to the boiling point of the solvent. Once the temperature was setup, the process was continued for 3 days till the colour change of the distillation column was observed. For 3 days, the boiling range of the solvents was maintained with proper condensing process of water in and out. After the colour change was observed the heating mantle was switched OFF and the distillation flask was separated to extract the oil from solvent. The solvents were recovered by distillation process, by heating the distillation flask to the solvent boiling temperature. Once the solvents are recovered, the oil was used for further analysis and conversion processes.

IV. PHYSICOCHEMICAL ANALYSIS OF JATROPHA OIL

Density analysis

The density of the extracted oil was determined to measure its efficacy in the conversion of free fatty acid to Biodiesel. Once after the recovery of solvent, the extracted oil was collected in the bottom flask. Then the oil was heated up using the heating mantle to remove the additional solvents present along with the oil. After the solvents are evaporated the mass of the oil was determined. To measure the mass, initially the empty flask was weighed followed by weighing the flask along with the oil sample. From that the weight of the oil is calculated as,

Mass of oil = weight of flask with the oil – weight of the empty flask

After that, the volume of the oil was measured using the measuring cylinder. Once the parameters were measured the density of the oil was determined using the mathematical calculation.

Determination of Saponification value

The determination of the acid value includes a typical process; known amount of sample was dissolved in an organic solvent and titrated against the solution of a base catalyst of known concentration with phenolphthalein as a color indicator. The acid value number must be less than unity. If the value exceeds, the pre-esterification process was carried out to reduce the acid value number. Mathematically, the value was determined by

Acid value number = [(A-B)*N*39.99]/W

0.1N of NaOH was prepared by dissolving 4g of NaOH in 1000ml of distilled water. Weighed 1g of the extracted oil and add 5ml of fat solvent (Ethanol) to the oil, mixed well and transferred to the conical flask. Again add 5ml of Ethanol to rinse well the container and transferred to the conical flask. The conical flask is mixed well to dissolve the oil completely to the added solvent and labeled as Test. Then, 25ml of 0.5N NaOH was added to the conical flask of Test. The blank was prepared by adding 10ml of the fat solvent (Ethanol) and 25ml of 0.5N NaOH in a separate conical flask. Both the test and blank flask are warmed under reflux condenser for 30mins and later allowed to cool. Add 1 to 2 drops of phenolphthalein color indicator to both the conical flask and the color changed to pink color. Then the content of the flask are titrated against 0.5N of HCl taken in the burette. The end point of the titration was indicated by the decolorisation of the pink solution in the conical flask.

V. CONVERSION OF JATROPHA OIL TO BIODIESEL

About 1L of the extracted oil (Jatropha seed oil) was taken in beaker and filtered to remove the unwanted particles, debris in the oil. Then the oil was heated using heating mantle above 100^{o}C to remove the additional water molecules present in the oil. The oil was pre-treated, and heated to a temperature of 60^{o}C under continuous stirring using a magnetic stirrer. The base catalyze solution was prepared by dissolving 4g of NaOH to 300ml of Methanol under magnetic stirrer over 30mins. Once the oil reached the temperature the base catalyze solution was added to the oil under constant stirring. After the solution was added the oil gets mixed well with the mixture and allowed to stir over a period of time. Then the mixture was transferred to the separating funnel and allowed undisturbed for over 24 hours. After 24hours, the layers get separated and the biodiesel was collected. The biodiesel was purified by washing with hot water to remove the additional catalyst present with the diesel. The glycerine obtained was used for further soap or foam production product. Collected Biodiesel was used for further analysis of Flame test, Thin Layer Chromatography and GC-MS analysis.

VI. CHARACTERIZATION OF BIODIESEL

Flame test

The first and basic confirmatory test for the biodiesel was the flame test which determines the presence of FAME (fatty acid methyl ester) components involved in the emission process in engines and machineries. The fatty acid methyl esters are known as the Green diesel, which was a renewable diesel derived from the biomass that does not contain monoalkyl esters of fatty acids. The flame test was carried out by burning the biodiesel in the presence of the flame.

Thin Layer Chromatography (TLC)

In TLC, stationary phase is a polar absorbent and the mobile phase can be a single solvent or combination of solvent. TLC is a quick, inexpensive microscale technique. Once the Samples were separated, they were visualized under the UV spectrometer or visualized by staining Sulphuric acid or by staining with Iodine solution. In addition to qualitative results, TLC can also provide a chromatographic measurement known as an Rf value. The Rf value was known as the “Retention factor” or the “ratio-in-front” value expressed as a decimal fraction.

Gas Chromatography-Mass Spectrometry (GC-MS)

The GC-MS was an analytical method that unites the features of both Gas-chromatography and Mass-spectrometry. GC-MS was mainly involved in the identification of different substances present within the Biodiesel sample by separating and quantifying. The instrument works in a way that where the gas chromatography (GC) portion involved in the separation of the chemical mixtures into pulses of pure chemicals and the mass spectrometer (MS) involved in the identification and quantify the chemical substances.

VII. RESULTS

Extraction of oil under individual solvents

After the 3 days of extraction process using the individual solvents of Hexane, Isopropyl alcohol and Petroleum ether under their optimum temperatures the oils were extracted as follows in the Table 1.

Table 1 Extraction of oil under individual solvents

S.No	Jatropha Seeds (g)	Solvent Name	Vol. of Sovent (ml)	Temperature (^oC)	Extracted Oil (ml)
1	50	Hexane	200	68	32
2	50	Petroleum Ether	200	62	17
3	50	Isopropanol Alcohol	200	82	28

Extraction of Oil under combination of solvents

The combination of solvents has increased the oil extraction when compared to the use of individual solvents that are recorded as given in the Table 2.

Table 2 Extraction of Oil under combination of solvents

S. No	Seeds (gm)	Solvents Name	Vol. of Solvent (ml)	Temperature (^oC)	Oil extracted (ml)
1	50	Hexane : Isopropyl alcohol	100: 100	82	14
2	50	Hexane : Petroleum ether	100: 100	68	20
3	50	Petroleum ether : Isopropyl alcohol	100: 100	82	30
4	50	Petroleum ether : Hexane : Isopropyl alcohol	66: 66: 66	82	32

Density of the extracted Oil

The density determines the lubricant only ensures the functions of machine performance; the change in the density changes the efficiency of the pump. Thus, density of the oil extracted from the different solvents were calculated and discussed in the Table3.

Table 3 Density of the extracted Oil

No	Seed (g)	Solvent Name (ml)	Extracted Oil (ml)	Density (kg/m^3)
1	50	Hexane	32	0.56
2	50	Isopropanol alcohol	28	0.62
3	50	Petroleum ether	17	0.77
4	50	Hexane: Isopropanol alcohol	14	0.64
5	50	Hexane: Petroleum ether	20	0.65
6	50	Petroleum ether: Isopropanol alcohol	30	0.45
7	50	Hexane: Isopropanol alcohol: Petroleum ether	32	0.51

Acid value determination of extracted Oil

The acid value measures the average molecular weight of all fatty acids present within the oil. The longer the chain of the fatty acid, lower the value of saponification number and vice-versa. Thus all the acid value determined for the fatty acids are lesser than unity, which increase the yield of biodiesel. The acid values are discussed in the Table 4.

Table 4 Saponification Analysis

No	Oil Extracted From Solvents	Acid Value Number
1	Hexane	0.43
2	Isopropanol Alcohol	0.412
3	Petroleum Ether	0.42
4	Hexane: Petroleum Ether	0.466

5	Hexane: Isopropanol Alcohol	0.421
6	Petroleum Ether: Isopropanol Alcohol	0.475
7	Hexane: Petroleum Ether: Isopropanol Alcohol	0.487

Thin layer chromatography (TLC)

The dark brown colour over the plates, confirmed the presence of the diesel compounds like methyl esters and ethanol of fatty acids. This shows that the produced biodiesel contains all the fatty acid compounds that are required for the emission of engines and vehicles (Figure 1).

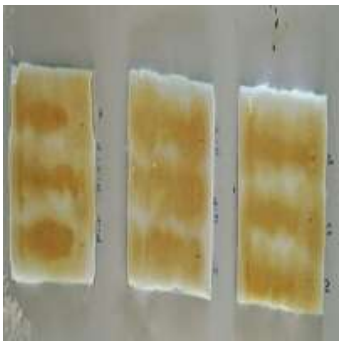

Figure 1 TLC for Biodiesel

Gas Chromatography Mass Spectrometry (GC-MS)

The quantification of Fatty Acid methyl Esters (FAME) that are present in the *Jatropha curcas* oil biodiesel was categorized by using the Gas Chromatography – Mass Spectroscopy. The major FAME's components that are present in the Jatropha oil Biodiesel are depicted below based on their Molecular weights and Retention values in the Figure 2.

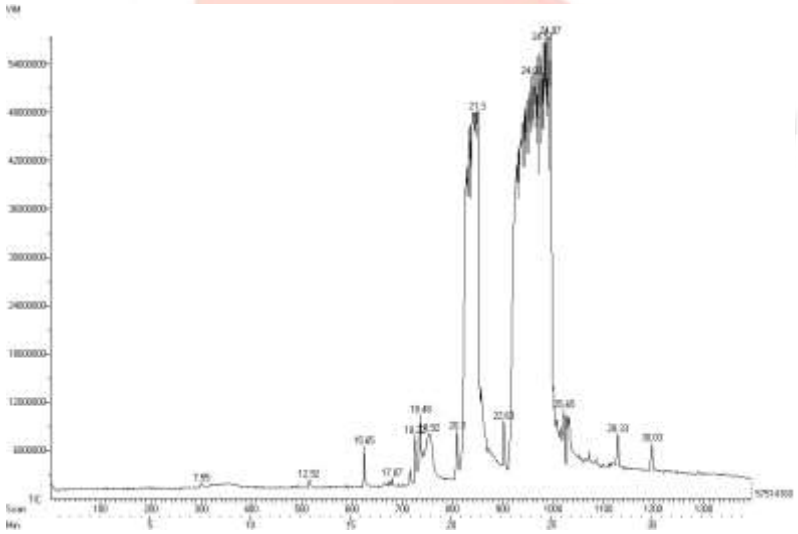

Figure 2 GC-MS Spectrum of *Jatropha curcas* oil Biodiesel

Conversion of oil to biodiesel

The diesel obtained was calculated to be of 980 ml from the use of 1000 ml of Jatropha Oil. This shows about 98% conversion of oil to biodiesel. Compared to the latest work of biodiesel production, the results shows the increased conversion rate of product from 95% to 98%. Thus, biodiesel production under the soxhlet extraction method gives a higher yield of product called Biodiesel.

VIII. SUMMARY AND CONCLUSION

The present work was carried out to investigate the optimization and characterization to increase the production of biodiesel by the plant source material of *Jatropha curcas*.

From the combinations of solvent the pair of ratio, Hexane: Petroleum ether: Isopropyl alcohol gave the best result for the Oil extraction that was equivalent to the Oil extraction of Hexane. This showed in limitation of Hexane, this pair of ratios can be used as alternative source for the production of oil. Similarly, in absence of Hexane either Isopropyl alcohol or the pair of ratio Petroleum ether: Isopropyl alcohol can be used as the best solvent in the extraction process for more yield of oil compared to Hexane. In individual solvent, Isopropyl alcohol can be used as the best substituent instead of Hexane as solvent.

From the Physico-chemical characterization, the TLC analysis preliminarily confirms the presence of methyl ester groups present in the biodiesel by visualization of TLC plates. The GC-MS analysis of the biodiesel identified the various fatty acid groups present within the biodiesel called the FAME along with their chemical compositions. The presence of 15 different fatty acids within the FAME showed the optimized method of biodiesel production that was more efficient in the future use of biodiesel production with the product conversion of 98%. Thus, Biodiesel can be used as an alternative fuel to reduce the load on conventional fossil fuel resources.

The future use of biodiesel not only limits the use of fossil fuel but also helps in the reduction of harmful air pollutants that are released during the combustion of conventional fuels, which therefore mentioned to be Green-fuel.

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