Production and Characterization Studies of Biodiesel from *Madhuca longifolia*

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Abstract-Nowadays world is suffered by the twin crisis of fossil fuel depletion and environmental degradation. We need to produce an alternative fuel that must be readily available and environmental friendly. BIO-DIESEL from Mahua oil has high potential to become alternative fuel to replace diesel fuel. Initially the Mahua oil has high viscosity which cannotbe directly used in the diesel engines. There are different methods to reducethe viscosity of Mahua oil. In this project trans esterification process is usedto reduce the viscosity of oil. Because of high free fatty acids in Mahua oil two step acid-base catalyst transesterification processes is used for producing the BIO-DIESEL. And the results based on physical properties of BIO-DIESEL produced from Mahua oil were close to the properties of diesel and efficiency test will be carried out.

Keywords: ***Madhuca longifolia,*** **Bio diesel, transesterification, Mahua oil,Viscosity.**

I.INTRODUCTION

The major percentage of energy used in the world today are being generated from fossil fuels are non-renewable resources that take millions of years to form and their reserves are being depleted faster than they are being regenerated. They are the major contributors and sources of greenhouse gases air pollution and global warming. Some of the emissions generated from these fossilfuels are CO, CO_2, NO_X, SO_X, un-burnt or partially burnt HC and particulate. Theproduction and use of these fossil fuels are raising environmental issue is seriously calling for an alternative.

Alternative fuel such as vegetable oils can use as sustainable fuels. There are several advantages of using vegetable oils as fuels such as higher energy content than other energy crop like alcohols. The vegetable oils have 90% energycontent of diesel. The current prices of vegetable oils in world are nearly competitive with petroleum fuel prices but these are not economically feasible yetand need further R&D work for development work. Vegetables oils can be successfully used in CI engine through engine and fuel modification. Engine modification includes dual fuelling injection modification system, heated fuel lines and blending of vegetable oil with diesel.

Large amounts of air pollutants, such as sulfur and solid carbon particles, are produced by diesel fuel, especially traditionally. To reduce greenhouse gas emissions, additional filtration steps and emission control mechanisms are implemented, which can turn it into a better fuel. Also, diesel fuel can produce more carbon dioxide than gasoline per unit.

1.1.*Madhuca longifolia*

The Mahua tree, or madhuca longifolia is grown in the wooded plains of western, central and eastern India, especially where tribes like the Santhals, Gonds, Mundas and Oraons live. These tribes have been living in this area for the last 3,000 years. They consider Mahua to be the "tree of life".The mention of Mahua is traced in ancient texts (Hinduism), and its wine was praised. Kalidasa, a poet from the fourth century, wrote about Goddess Parvati wearing its flowers as a garland. However, its culture is even older, and it is most ingrained in India's tribal interior, where people have resisted joining the Hindu mainstream.

Mahua oil (*Madhuca Longifolia*) is non-edible oil available in huge quantities in south Asia. Annual production of Mahua oil in India is estimated tobe 60 million tonnes. Traditionally it has been used as fuel in lamps for lightening purposes in rural areas and is used on industrial scale for manufacturing of soaps, detergents, vegetable butter and other non-edible products. Mahua oil is used to make a variety of formulations that aid in treatingailments. The seed on Mahua yield 30 % - 60 % weight of kernel on dry matter basis is non-edible oil that can be used in BIO-DIESEL production. It grows in all kinds of soil and in drier areas. Mahua tree is an evergreen tree growing in almost every state of India. It is a multipurpose tree native to Indian subcontinent and south- East Asian countries and it is a cultivated all over India. It grows in dries areas and in all kinds of soil. Itcontains several thousands of chemicals which are Terpenoids in nature. A mature Mahua tree produce 30 to 50 kg fruit every year. It has a productive life span of 150 to 200 years. Mahua oil is derived from crushing theMahua seed and by using mechanical expellers.

The aim of this study deals with to produce an alternative and ecofriendy source for diesel by using *Madhuca longifolia* and its biological and chemical derivative analysis.

II. MATERIALS AND METHODS

2.1 Sample Collection

Mahua seeds were purchased from the Thangam oil mill near Aasaripallam.. Mahua seeds were used for the extraction of oil by oil expeller and solvent extraction method and biodiesel production by transesterification reaction and its quality analysis.

2.2 Phytochemical analysis

A qualitative phytochemical test to detect the presence of alkaloids, tannins, saponins, flavonoids, glycosides and phenols. The intensity of the coloration determines the abundance of the compound present. Ethanolic and methanolic extract of Madhuca longifolia flowers were subjected to preliminary phytochemical study following the methodology of Kokate, Purohit and Gokhale. Ethanolic extract of Madhuca longifolia showed the presence of alkaloids, tannins, proteins, carbohydrates. Methanolic extract showed the presence of alkaloids, tannins, carbohydrates

2.3 Biochemical Properties

Specific Gravity

The Mahua oil which was extracted from oil expeller was filled in 500 ml measuring cylinder and the hydrometer was dipped in the oil and the density of oil was recorded. The specific gravity of the oil was calculated by using the following formula: Specific gravity= density of oil /density of water.

Free fatty acid (FFA) content (AOAC, 2000)

The Mahua oil which was extracted from the oil expeller was titrated by using 0.1 N NaOH freshly prepared solution in a clean burette. Simultaneously, 30ml of isopropanol was taken in a clean 250ml conical flask and 3-4 drops of 0.1 N NaOH (neutralization) and then 10gms of oil sample was added to the flask and then heated to 500C and allowed the solution to cool and then 5-6 drops of phenolphthalein indicator was added and titrated against the 0.1 N NaOH until the colour turns from yellow colour to permanent pink colour (Doddabasawa et al., 2014)

2.4.Biodiesel Production

Mahua biodiesel was prepared using two step processes. Crude unrefined mahua oil was brownish yellow in colour.

I)Transesterification process: a) Preheating of esterified oil: b) Transesterification process using alkaline catalysis c) Separation of Glycerol from Transesterified oil (FAME) d) Neutralizing Transesterified oil (FAME)

II) Acid esterification: a) Preheating of raw oil b) Esterification process using acid catalysis c) Separation of Methanol from Esterifies oil d) Neutralized Esterifies oil Qualitative analysis of Biodiesel

2.5 CHARACTERIZATION STUDIES

2.5.1 FTIR:-

Fourier Transform Infrared (FTIR) analysis was done for to identify the functional groups .FTIR spectrum was applied under transmittance mode.

2.5.2 GCMS Analysis

Qualitative and quantitative GC/MS analyses were done in Mahua oil.

2.5.3 Thin layer chromatography (TLC)

Silica gel slurry was prepared using 0.02M sodium acetate buffer and spread on TLC plate having 250µm thickness. The TLC plate was activated by heating at 105o C for 30 minutes. 5μl of mahua seed oil sample extracted and olive oil as a standard for comparing the Rf values which were spotted on the TLC plates. The spots were dried in air and were kept in a TLC chamber containing Petroleum ether: Diethyl ether: Acetone (90:10:1). TLC plates were kept for lipid components separation for around 30-40 minutes and the spots were visualized by spraying the 0.2% Ninhydrin reagent and then the Rf values were calculated.

III. RESULTS AND DISCUSSION

3.1 Mahua Oil Production

Mahua oil seed collection and oil production at Thangam oil mill near Aasaripallam.

Fig No.1 Mahua seed, Fig No.2 Mahua oil

3.2 Acid Esterification

BIO-DIESEL produced by transesterification reaction can be catalyzed by sulphuric, phosphoric, hydrochloric and organic sulfonic acids. Currently the catalysts more used in BIO-DIESEL production are the organic acids such as the derivates of toluene sulfonic acid and, more often mineral acids such as sulphuric acid. Although transesterification using acid catalysts is much slower than that obtained from alkali catalysis, typically 4000 times, if high contents of water and FFAs are present in the vegetable oil acid catalyzed transesterification can be used. The FFAs react with alcohol and directly converted into BIO-. DIESEL

Fig. No.2 Separation of oil and glycerol, Biodiesel

3.3 Crude Bio-Diesel:

After separation from the glycerol phase crude BIO-DIESEL is contaminated with catalyst, water, and unreacted alcohol, glycerol and soaps these are generated during transesterification process. The contaminated materialsshould be removed before using in the engines. These contaminants can remove using water washing process.

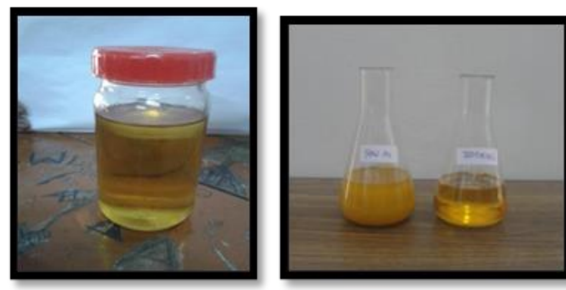

Fig. No.3a Collected Biodiesel, b) Raw oil and biodiesel

3.4 Phytochemical analysis

A qualitative phytochemical test to detect the presence of alkaloids, tannins, saponins, flavonoids, glycosides and phenols. The intensity of the coloration determines the abundance of the compound present. Ethanolic and methanolic extract of *Madhuca longifolia* flowers were subjected to preliminary phytochemical study following the methodology of Kokate, Purohit and Gokhale. Ethanolic extract of Madhuca longifolia showed the presence of alkaloids, tannins, proteins, carbohydrates. Methanolic extract showed the presence of alkaloids, tannins, carbohydrate.

Table1 –Phytochemicals

phytochemicals	Result
Alkaloids	+
Tannins	+
Proteins	+
Flavonoids	-
Carbohydrates	+
Amino acids	-
Volatile oils	-

(+)-Positive,(-)-Negative

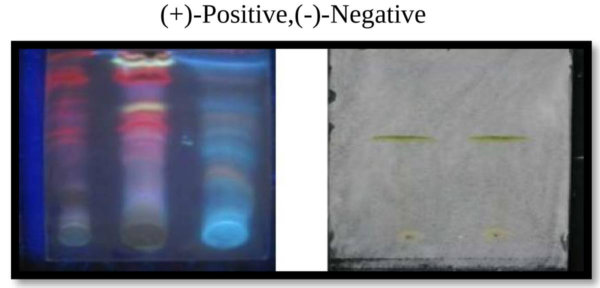

Fig. No.4 TLC Results

3.5 CHARACTERIZATION STUDIES

3.5.1 FTIR:-

Fourier Transform Infrared (FTIR) analysis was done for to identify the functional groups .

FTIR spectrum was applied under transmittance mode in the wavenumber range from 500 cm^{-1} to 4000 cm^{-1}.Sharp prominent peaks are obtained at 2920 cm^{-1} and 2826 cm^{-1} due to-C-H stretching, which corresponds to calcium groups.The peaks at 1721 cm^{-1} and at 1475 cm^{-1} are due to carbonyl stretching ofthe ester functionality. The presence of carboxyl group was revealed by the peaks obtainedat stretching frequencies at 1350 cm^{-1} and 1168 cm^{-1}.

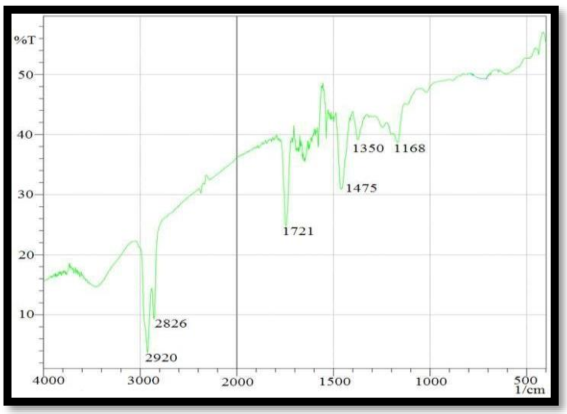

Fig. No.5 FTIR Analysis

3.3.2 GCMS Analysis

Qualitative and quantitative GC/MS analyses were done in Mahua oil.Particular chemicals were identified

3.3.3 TLC – Thin Layer Chromatography for Mahua seeds

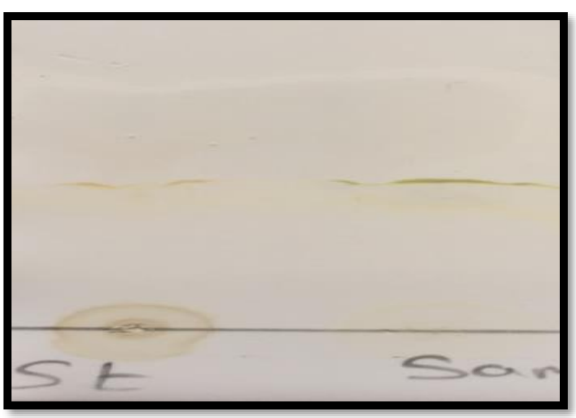

Fig. No.6 TLC for Phytochemicals

Fig 3 TLC – Thin Layer Chromatography for Mahua seeds

3.4 ACID VALUE OF RAW MAHUA OIL

Acid Value (mg KOH / g) =(Titrate value × Normality of KOH×56.1) /

The high FFA (8%) content Mahua oil has been investigated for the BIO-DIESEL production. It has been found that the feedstock with high FFA could not be trans esterified with base catalyst. The reason is base catalyst react with FFA to form soap. In order to reduce high FFA two-step process was developed to convert FFA to its methyl ester. The first step is acid treatment which reduces the FFA content of oil to less than 2%. The second step is base catalysed reaction. The effect of molar ratio was carried out in each step and the optimum parametersfor acid catalysed trans esterification reaction are 1% H_2SO_4, 9:1 (Methanol: oil) ratio at 55˚C temperature and 60 min reaction time the yield is 96%. For base trans esterification reaction 1% KOH, 6:1 (Methanol: oil) at 55˚C and 120 min reaction time the yield is 89%. The properties such as density, Viscosity, Flash point, Calorific value, Acid value, Phosphorus content, Sulphurcontent, Carbon residue, Iodine value, Saponification value and Cetane number and FFA were find out and which are comparable to diesel and which can be used in diesel engines.

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