# **Biodiesel Production from Pongamia Pinnata and its Characterization using GC-MS, NMR and FT-IR Spectral Studies**

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## **ABSTRACT:**

Biodiesel synthesis from the pongamia oil seed and its characterization is elaborated in this paper. A double stage transesterification i.e. acid catalysed transesterification and base catalysed esterification are adopted to reduce the free fatty acids content and conversion of triglycerides into methyl esters. In this process, H<sub>2</sub>SO<sub>4</sub>, NaOH and methanol are used at the methanol/oil molar ratio of 7:1. By this process, 95% of pongamia biodiesel is obtained. The physiochemical properties like calorific value, Cetane number, density, kinematic viscosity, flash point, fire point etc. are analysed and it is found to be within the ASTM standards. GC-MS analysis indicated the existence of 14 prominent fatty acids with oleic acid as the major constituent. 13C and 1H NMR results supported the GC-MS data and it also confirmed the conversion efficiency of converting the vegetable oil into PBD as 87.23%. The shifting and appearance of major peaks in the FT-IR spectrum confirmed the formation of FAMEs from the triglycerides.

## **KEYWORDS:**

Biodiesel; Pongamia pinnata; Transesterification; GC-MS; FT-IR; NMR

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# 1. Introduction

Ever increasing energy demand, increase in petroleum fuel pricing and depletion of fossil fuels along with hazardous environmental pollution necessitated to explore various alternate possibilities of fuelling the engine system. The energy requirement prediction in the year 2030 is estimated to be 50% more than the present scenario with India being the fourth largest energy consumer after China, USA, and Russia. India's fuel energy demand continues to increase in spite of the global slowdown. With 25% of its energy requirements is accounted by industrial sectors and 75% is consumed by the transportation sector. It is understood that the demand for fuel energy will continue to intensify with rise in economic factors in the near future. Compression Ignition (CI) engines are extensively used in heavy duty and medium applications as main power source due to its minimal fuel consumption, enhanced robustness and increased thermal efficiency [1-3]. Biodiesel is a biodegradable and non-toxic fuel.

Development of CI engines from the past was mainly based on diesel fuel. Diesel-biodiesel blend with comparable physiochemical properties can be used with minimal changes in operating and design parameters of the engines. Substantial investigations are being carried out by the researchers to identify an alternate fuel in place of the petroleum diesel having comparable physiochemical properties. Biodiesel from non-edible oil sources favoured its application as a fuel in CI engines. Non edible oil seeds such as cotton seeds, pongamia, neem, castor, mahua, Jatropha, linseed, rapeseed and others are being examined for its use as fuel in CI engines as straight and blended forms. In spite of rapid depletion of the fossil fuels and increase in the crude petroleum pricing, biodiesel from non-edible vegetable oil seems to be an encouraging substitute for fractional replacement of petroleum products. Production cost of the biodiesel can be brought down by choosing nonedible vegetable sources thereby averting energy-food competition to a very large extent.

Biodiesel produced through transesterification of vegetable oil with enhanced catalytic action is comparable with petro-diesel fuel. Generally, acid catalyzed and base catalyzed transesterification is adopted depending on the FFA level of vegetable oil. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) along with sulfuric acid  $(H_2SO_4)$  are the commonly used catalyst in the transesterification process. Preceding investigations reported the reduction of exhaust emissions especially HC with the usage of biodiesel [4-6]. Meher et al [7] adopted methanolysis process to derive biodiesel from pongamia pinnata oil by using KOH as catalyst. Optimal biodiesel production was determined at 65°C reaction temperature and molar ratio of 12:1. The characterization of biodiesel was carried out using HPLC and TLC to identify the various fatty acid methyl esters (FAMEs) [7]. Gopal and Raj [8] transesterified the pongamia oil using NaOH and prepared pongamia oil methyl esters.

PME (pongamia methyl esters) was blended with diesel at various proportions and their effect on the engine performance characteristics was studied. The variation in physiochemical properties of the lubricating oil like flash point, density, moisture content, total base number, ash content, kinematic viscosity, pentane and benzene insolubility, and concentration of metallic elements were studied [8]. Carmen et al [9] carried out a comparative study on animal fat biodiesel by partially the replacing diesel and soya bean biodiesel. The brake specific fuel consumption and brake thermal efficiency was increased along with significant rise in NO<sub>x</sub> emission. Also, it was found that animal fat biodiesel concentration beyond 25% reduced the particulate matter emissions significantly. Tariq et al [10] synthesized biodiesel from rocket seed oil through single stage basecatalyzed transesterification. Spectroscopic studies like FT-IR and NMR confirmed the presence of FAMEs in the biodiesel. The physiochemical properties of fuel was tested and found within ASTM standards. The GC-MS analyses revealed the presence of saturated and unsaturated FAMEs in the biodiesel.

Nabi et al [11] extracted oil from pongamia seed higher FFA contents. Double having stage transesterification was employed for the production of biodiesel and it was found to be within ASTM and EN standards. The FT-IR and GC-MS analysis revealed the presence of methyl esters at about 97%. On fuelling the CI engines, it was found that engine noise, CO, smoke drastically reduced along with a significant increase in NO<sub>x</sub>. Guzatto et al [12] optimized the transesterification parameters, condition and reaction mixtures. Two stage transesterification with homogenous catalyst was used in the study. Pure and quality enhanced biodiesel was derived by using ethanol as an optimized parameter. The result was followed with NMR, FT-IR and physiochemical analysis of the biodiesel.

Based on the above, the present investigation attempts to explore the possibilities of using Pongamia as biodiesel. pinnata seed oil Two stage transesterification processes are employed to reduce the free fatty acid content and convert the non-edible vegetable oil into FAMEs. Gas Chromatography-Mass Spectroscopy (GC-MS), Nuclear Magnetic Resonance (NMR) and Fourier Transform Infra-Red Spectrometry (FT-IR) analysis are carried out to understand the characteristics of the biodiesel. Physiochemical properties are also determined as per ASTM standards.

# 2. Materials and methods

Pongamia seeds are purchased from a local vendor in Padur, Chennai. The seeds are dried in sunlight from 72 hours and followed by washing it with distilled water to remove the impurities. The moisture content is removed by placing the seeds in an electrical oven kept at  $75^{\circ}$ C for 120 minutes. Oil expeller technique is adopted to extract the oil from processed pongamia seeds which yielded 91% of the vegetable oil content.

# 2.1. Acid catalysed transesterification

Titration method indicated a higher level of FFA content by up to 12% in pongamia seed oil. 1000 ml of pongamia seed oil is taken in a three-necked flat bottomed flask fitted with magnetic stirrer, sampling outlet and temperature indicator. The oil is preheated up to 70°C before the start of the chemical reaction. 200ml of methanol and 1.5% of  $H_2SO_4$  are mixed with oil in the flat bottom flask to initiate the reaction. The agitation speed is maintained at 450rpm with a reaction temperature of around 65°C for 60 minutes. Continuous monitoring of FFA content is carried out during the reaction process and subsequently the FFA content is bought down to 0.5% confirmed by the formation of a ring layer at the bottom of the flask. By this process, 93% of pre-treated pongamia oil is obtained.

# 2.2. Base catalysed transesterification

Based on the literature review, the maximum yield of biodiesel is achieved only with lower concentration of FFA. Higher concentration of FFA results in soapy sludge formation which affects the biodiesel production. One litre of pre-treated pongamia oil is taken in a round bottom flask fitted with a heater, magnetic stirrer, sampling outlet and temperature indicator. Sodium methoxide solution is prepared by adding 450ml of methanol with 0.25% of sodium hydroxide. 300ml of sodium methoxide solution is mixed with the pre-treated pongamia oil. The chemical reaction is maintained between 60°C to 75°C at an agitation speed of 450 rpm to ensure maximum conversion of triglycerides into FAMEs. The entire mixture is transferred into a separating funnel with a settling time of 24 hrs. During this settling period, the glycerol is settled at the bottom layer along with the formation of ring below the methyl esters of pongamia seed oil. The methanol oil molar ratio of 7:1 as indicated above is found to be the optimum range for biodiesel production. By this process 95% of pongamia biodiesel (PBD) is obtained. Further variations in the molar ratio and catalyst concentration yielded negative results in the formation of biodiesel [13].

# **3.** Characterization techniques

PBD produced through two stage transesterification processes is studied for its physio-chemical properties like density, kinematic viscosity, pour point, specific gravity, sulphur content, calorific value, Cetane number, flash point, fire point, acid content and cloud point. The properties are found to be comparable the ASTM standards as given in the Table 1. GC-MS analysis on PBD is performed using the JEOL GC MATE II data system fitted with a high resolution, double focusing facility. The maximum calibrated mass is 1500 Dalton with a maximum resolution of 6000. Helium acts as a carrier gas with a flow rate of 2 ml/sec. The biodiesel test sample was injected into the capillary column with a pre-programmed temperature setting of 150°C to 320°C. The scanning of GC-MS spectrometer is customized with an electron impact ionization mode kept at the range of 50 to 500 m/z. The vibrational motion of molecular compounds in the PBD sample is studied using the Perkin Elmer 1 FT-IR spectrometer with a scanning range of MIR 450-4000 cm-1. 50 mg of PBD is injected in order to obtain the various vibrational frequencies of the compound through FT-IR technique. The various functional groups like -CO, -OH, -CN, -CH and others are also estimated for the presence of FFA.

Table 1: Comparison of physiochemical properties of PBD and
commercial diesel

Property	PBD	Diesel	ASTM D6751	
Density at 30°C (kg/m <sup>3</sup> )	888.38	830	870-900	
Kinematic viscosity at 40°C	4.79	4.85	1.9-6.0	
Specific gravity	0.890	0.80	0.876-0.939	
Calorific value (kJ/kg)	38404.94	44800	35000-42000	
Cetane number	46	50	40-55	
Ash content (% by mass)	0.0085	-	-	
Sulphur content (% by mass)	0.002	-	>0.050	
Flash point (°C)	92	71	130- 196	
Fire point (°C)	103	82	-	
Acid value (mg/KOH)	0.42	-	>0.50	
Pour point (°C)	-3	-5	-4 to 15	
Cloud point (°C)	11	-1	-3 to 22	

NMR spectrometry system is equipped with Bruker AVANCE III 500 MHz multi nuclei solution spectrometer fitted with 11.7 tesla super conducting magnet actively shielded with cryo-shim, 34 channel shims and standard bore is used to characterize the PBD. Broadband gradient probe head with auto-tuning VT and quadruple inverse probe with gradient are used. The 13C and 1H NMR spectra are recorded with appropriate pulse duration. Deuterated chloroform is used as a solvent for injecting the biodiesel.

#### 4. Results and discussion

The detailed physiochemical properties of PBD along with ASTM biodiesel standards are given in Table 1. The density of PBD is found to be 888.38 kg/m<sup>3</sup> which are higher than the commercial diesel. The kinematic viscosity is noticed to be lower in PBD, but it is within the ASTM standards. A marginal increase in the specific gravity of PBD is noticed. The flash and fire point of PBD is noticed to be 92°C and 103°C respectively which is higher than the commercial diesel making it safer during storage and transportation. The cloud and pour point too is found to be within limits. The acid value of PBD is determined as 0.42 mg/KOH. FAMEs present in the PBD are identified by GC-MS analysis. The GC-MS chromatogram of PBD showed 15 major peaks. Each peak resembles unique FAMEs content present in the PBD. NIST 2 library software identified the various FAMEs present in the biodiesel. The FAMEs which are identified is confirmed with a library by comparing its retention time, fragmentation pattern under similar experimental condition. The GC-MS analysis revealed the existence of six unsaturated FFA and eight saturated FAMEs in the PBD.

Fig. 1 depicts the GC-MS of PBD. From the mass chromatogram, oleic acid presence is identified with prominent quantity at a retention time of 24.33. All the 14 FAMEs shared their base peak at m/z 74 which is due to Mclafferty re-arrangement process [15]. The fragmentation pattern identified the presence of FAMEs by comparing the other two peaks which is due to the lose methoxy/methylene ion and hydrogen atoms rearranged in the long chain. The loss of methoxy functional group is also termed as  $\alpha$ -cleavage. Also propyl radical expulsion is followed by the rearrangement of hydrogen atoms at m/z 180.  $\beta$ -cleavage resulted in the formation of carbo-methoxy ion at m/z 83, 97, 111, 137 and it is found in larger quantities in many regions. Thus,  $\alpha$ -cleavage,  $\beta$ -cleavage, carbo-methoxy ion and hydrogen atom rearrangement helped in distinguishing and confirming the presence of FAMEs in PBD. The various prominent FAMEs present in the PBD are palmitic acid, stearic acid, oleic acid, myristic acid, eicosanoic acid and arachidic acid at a retention time 14.73, 17.60, 20.32, 24.33, 25.65, 25.25 and 28.15 respectively. The details of various FFA present in PBD are tabulated in Table 2.

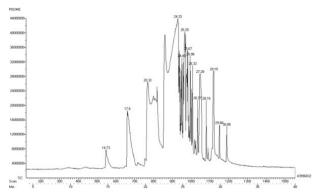


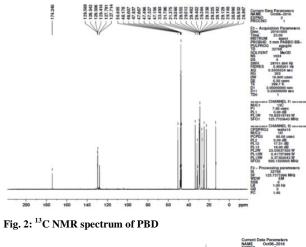
Fig. 1: GC-MS of PBD

Table 2: GC-MS analysis of PBD, ME = Methyl Ester

Peak No	RT	Ester description	Acid name
1	14.73	Dodecanoic acid ME	Lauric acid
2	17.60	Methyl tetradecanoate	Myristic acid
3	20.32	Hexadecanoic acid ME	Palmitic acid
4	24.33	10-octadecenoic acid ME	Oleic acid
5	24.85	10, 13 Eicosadienoic ME	Eicosanoic acid
6	25.25	11 Eicosanoic ME	Eicosanoic acid
7	25.67	Isopropyl stearate	Stearic acid
8	25.98	9-Octadecenoic acid (Z) - 2- butoxyethyl ester	Oleic acid
9	26.97	13-Docosenoic acid ME	Erucic acid
10	27.28	Docosanoic acid ME	Behenic acid
11	28.15	Tricosanoic acid ME	Arachidic acid
12	29.15	Tetracosanoic acid ME	Lignoceric acid
13	29.88	Pentacosanoic acid ME	Oleic acid
14	30.85	Hexacosanoic acid ME	Cerotic acid

## 4.1.NMR

Fig. 2 represents the characteristic spectrum of Carbon (<sup>13</sup>C) NMR of the PBD. Strong signals at 47.667 ppm and 2.192 ppm represent the major peaks of carbonyl group and methyl esters. Minor peaks between 127.714 ppm and 129.589 ppm indicate the presence of unsaturated compounds comprising FAMEs. A terminal peak is also noticed at 16.230 ppm which is mainly due to the long chain hydrocarbon of the methylene group presence in the PBD. <sup>1</sup>H NMR spectroscopic analysis is used to characterize PBD as shown in the Fig. 3. A singlet is observed at 3.633 ppm along with multiple triplets at 2.321 ppm and 1.620 ppm. These are the characteristic peak due to the presence of methoxy protons and  $\alpha$ -methylene proton respectively. The characteristic singlet and triplet peaks confirm the presences of FAMEs in the PBD. Terminal peaks are also noticed at 4.843 ppm which is due to methyl protons. A strong signal is noticed at 0.932 ppm as a result of chemical shifting due to  $CH_2$  proton in the hydrocarbon chain. Integrated signals due to methoxy proton group and carbonyl methyl proton group are noticed at 3.369 ppm and 2.306 ppm which confirms the existence of methyl esters in the PBD. Generally, proton NMR is used to analyse the quantity of vegetable oil conversion into FAME through transesterification process. The transesterification efficiency in converting the pongamia vegetable oil into its corresponding biodiesel is found to be 87.23% which is comparable with the theoretical conversion rate of 89.23%. The yield of biodiesel can be increased by augmenting the reaction time and settling period along with advanced strategies for biodiesel-glycerol separation [14-16].



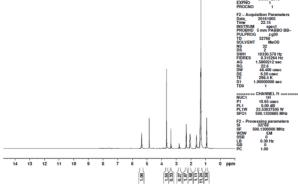


Fig. 3: <sup>1</sup>H NMR spectrum of PBD

#### 4.2. FT-IR spectrometry

The various functional groups existing in the mid-range of the FT-IR spectrum and their corresponding bandwidth along with the bending and stretching vibration for pongamia oil and its biodiesel are investigated to analyze the presence of FAMEs as shown in Fig. 4. Two characteristics strong signal due to presence of carbonyl group and esters are observed between stretching vibration 1820-1670cm<sup>-1</sup> and 750-1735cm<sup>-1</sup>. Also, it is observed at 1118-1010cm<sup>-1</sup> due to asymmetrical stretching vibrations. Higher stretching vibration is observed between 3200-2700cm<sup>-1</sup>. Stretching vibrations of alkanes, alkynes, amines appeared at 3000-2910cm<sup>-1</sup>, 2330-2090cm<sup>-1</sup> and 1215-1080cm<sup>-1</sup> whereas, bending vibrations is observed at 1370-1350cm<sup>-1</sup> and 1685-1605cm<sup>-1</sup>. The appearance of peaks at 1435cm<sup>-1</sup>, 1361cm<sup>-1</sup>, 1245cm<sup>-1</sup>, 1170cm<sup>-1</sup> and 722cm<sup>-1</sup> along with

disappearance of peaks at 1875cm<sup>-1</sup>, 1721cm<sup>-1</sup>, 1216cm<sup>-1</sup>, 911cm<sup>-1</sup> and 890cm<sup>-1</sup> indicated the formation of PBD. It can also be noticed that the peaks 1459cm<sup>-1</sup>, 1118cm<sup>-1</sup> and 1017cm<sup>-1</sup> was shifted from 1472cm<sup>-1</sup>, 1072cm<sup>-1</sup> and 997cm<sup>-1</sup> confirming the existence of the FAME [18]. FT-IR mass spectrum for pongamia oil and its biodiesel are almost identical. The appearance and shifting of the major peaks confirmed the transformation of triglycerides into di-glycerides and mono glycerides during the transesterification process [17].

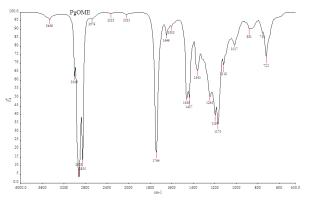


Fig. 4: FT-IR spectrum of PBD

#### 5. Conclusions

This study dealt with the production of PBD from the pongamia oil seeds and its characterization through a two stage transesterification process. Acid catalyzed transesterification with H<sub>2</sub>SO<sub>4</sub> and methanol reduced the FFA contents to less than 2% and it is followed by single base-catalyzed esterification. stage NaOH and methanol/oil molar ratio of 7:1 yielded 95% of PBD. The GC-MS analysis publicized the presence of 14 prominent FAMEs ranging between  $C_{13}$  to  $C_{27}$  in which oleic acid is found to be the primary constituent. This is verified by the analysis of fragmentation patterns and the retention time. The appearance and shifting of string vibrational signal between 2330cm<sup>-1</sup> and 1600cm<sup>-1</sup> indicated the conversion of triglycerides into FAMEs.

In <sup>13</sup>C NMR, strong signals at 47.867 ppm and 2.192 ppm represented the carbonyl group and methyl ester formation. The appearance of strong singlet at 3.633 ppm in <sup>1</sup>H NMR is due to the methoxy proton which confirmed the presence of methyl esters in PBD. The kinematic viscosity of PBD is found to be lower than the commercial diesel. The density, Cetane number, calorific value, flash point and fire point of PBD are found to be comparable with the mineral diesel and it is within the ASTM standards. Higher flash and fire points of PBD indicated its potential for safer storage and transportation compared to commercial diesel. Based on these, it can be concluded that PBD is one of the biodiesel which could be used as a substitute for the fossil fuel.

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