Production of biodiesel from non-edible *Pongamia Pinnata* feedstock using transesterification and nanocatalysts

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Abstract: The search for alternatives to petroleum-based fuels has led to the development of renewable fuel sources derived from fats and oils. Biodiesel, a prominent biofuel, is produced from triacylglycerol-rich feedstocks such as Karanja oil. This study explores the production of biodiesel from Karanja oil using a two-step process: acid-catalyzed esterification followed by nano-catalyst transesterification. Initially, raw Karanja oil is preheated and mixed with ethanol in a 1:8.5 and 1:9 molar ratio, treated with p-toluenesulfonic acid (PTSA) (3.2% w/w), and heated at 62°C for two hours to produce esterified oil. The transesterification process is then carried out using a nano-CaO catalyst (1.21% w/w) with an ethanol-to-oil molar ratio of 1:8.5 under continuous stirring (330–380 rpm) for two hours. The reaction yields trans-esterified oil, followed by phase separation to remove glycerol, resulting in biodiesel yields of 92% and 88%. Purification through multi-washing with deionized water enhances the final biodiesel yield to 73%. The biodiesel is blended with diesel at a 5% ratio, complying with industry standards and Indian biodiesel policy. Additionally, engine performance is evaluated at blend ratios of 5%, 10%, and 20%. The study provides insights into biodiesel production efficiency and its potential as a sustainable fuel alternative.t.

Keywords: Biodiesel, Pongamia Pinnata, Alternative Fuel, Transesterification.

I. INTRODUCTION

Global energy consumption has been on a continuous rise for decades, except for brief periods like the oil crisis in the 1970s, which temporarily slowed growth (Ahmad, Barat, Mohammad Reza, & Gholamhassan, 2012). In 2014, energy consumption increased by more than 5% following a slight decline in 2013. The rapid growth of the global population and industrial sectors has led to an increasing demand for energy, causing resource depletion and significant environmental pollution [2] [1]. The automobile sector has experienced tremendous expansion over the last two decades, resulting in higher fuel consumption and increased emissions. Diesel engine pollutants pose serious threats to both the environment and human health.
Biodiesel, [3] a renewable and sustainable alternative to fossil fuels is produced through the transesterification of edible and non-edible plant-based oils, such as vegetable oils and tree-borne oil seeds. [4] Transesterification is a reversible catalytic reaction that occurs by mixing the reactants under suitable conditions [5] [6]. Currently, biodiesel production relies heavily on traditional chemical catalysts. However, [7] advancements in nanotechnology present new opportunities for improving biodiesel production efficiency [8]. This study explores the potential of Kararja as a biodiesel feedstock using nano-catalysts and examines its impact on sustainability.

A. *Optimal Findings and Literature reviewed*

The high cost of biodiesel, primarily due to feedstock expenses (around 80% of operating costs) [1], limits its commercialization. However, blending ethanol with biodiesel (BE20) has shown promise in mitigating operational issues in unmodified diesel engines while reducing exhaust emissions and maintaining 90% renewable content [9]. Selecting an optimal feedstock is complex, requiring considerations like availability, oxidation stability, performance, and low-temperature operability [10]. While biodiesel can be used in diesel engines at low blend ratios (5–10%) without modifications, higher blends require further research [11]. The transesterification process, influenced by factors such as molar ratios, catalysts, temperature, and reaction time, typically follows a 6:1 alcohol-to-glyceride molar ratio, with base catalysts (0.1–1% w/w) proving the most effective [12]. Supercritical alcohol transesterification methods have demonstrated high efficiency, with conversion yields rising from 50% to 95% within 10 minutes and reaching 96% using a non-catalytic supercritical methanol process [13]. Biodiesel derived from *Pongamia pinnata* with methanol and KOH catalyst achieved a 92% conversion at a 1:10 molar ratio and 62°C, increasing to 95% with tetrahydrofuran as a co-solvent, while its fuel properties met ASTM and German standards [14].

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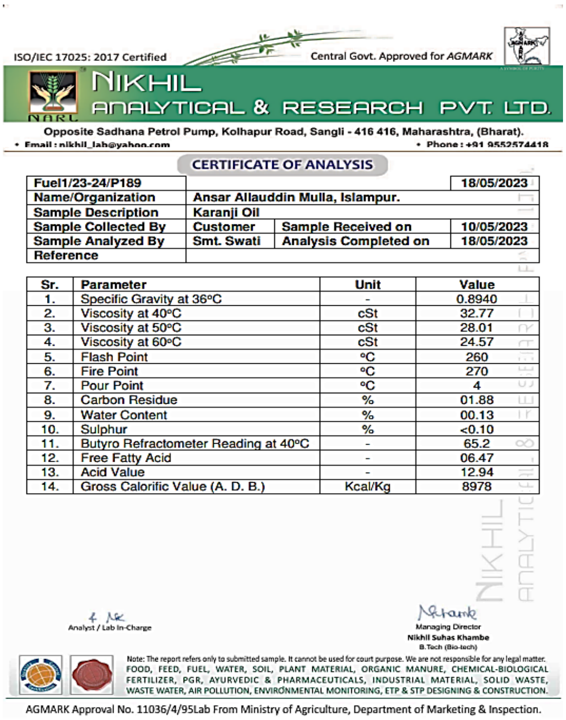
A 20% biodiesel blend with 15% exhaust gas recirculation (EGR) was found optimal for improving thermal efficiency, reducing emissions, and lowering brake-specific energy consumption (BSEC), effectively reducing NOx emissions without increasing smoke emissions [15].

II. METHODOLOGY

The paper depicts the nonedible feedstock its characterization and the production process followed using the optimal method, transesterification. The parameters for transesterification are studied and finalized from the earlier optimized research papers and the combinations are studied. The maximum yield with transesterification and nanocatalysts Cao and PTSA is used for biodiesel production from *P. Pinnata* oil.

B. *Characterization of feedstock for Biodiesel production*

The characterization of feedstock is a crucial step in biodiesel production, as the physical and chemical properties of the raw material directly influence the yield, quality, and efficiency of the transesterification process. Key parameters assessed for Karanja oil or any biodiesel feedstock include free fatty acid (FFA) content, viscosity, density, moisture content, saponification value, and iodine value. High FFA content can lead to soap formation during base-catalysed transesterification, necessitating a pre-treatment step such as esterification. Viscosity affects the flow and atomization of biodiesel in engines, while density influences fuel combustion characteristics. Moisture content must be minimized as water presence can cause hydrolysis of triglycerides and catalyst deactivation. The saponification value indicates the average molecular weight of fatty acids in the oil, and the iodine value determines the degree of unsaturation, affecting oxidative stability. Proper feedstock characterization ensures optimal processing conditions, improved biodiesel quality, and compliance with fuel standards, making it a vital aspect of biodiesel production.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CERTIFICATE OF ANALYSIS

Fuel1/23-24/P189			18/05/2023
Name/Organization	Ansar Allauddin Mulla, Islampur.		
Sample Description	Karanji Oil		
Sample Collected By	Customer	Sample Received on	10/05/2023
Sample Analyzed By	Smt. Swati	Analysis Completed on	18/05/2023
Reference			

Sr.	Parameter	Unit	Value
1.	Specific Gravity at 36°C	-	0.8940
2.	Viscosity at 40°C	cSt	32.77
3.	Viscosity at 50°C	cSt	28.01
4.	Viscosity at 60°C	cSt	24.57
5.	Flash Point	°C	260
6.	Fire Point	°C	270
7.	Pour Point	°C	4
8.	Carbon Residue	%	01.88
9.	Water Content	%	00.13
10.	Sulphur	%	<0.10
11.	Butyro Refractometer Reading at 40°C	-	65.2
12.	Free Fatty Acid	-	06.47
13.	Acid Value	-	12.94
14.	Gross Calorific Value (A. D. B.)	Kcal/Kg	8978

Analyst / Lab In-Charge

Managing Director
Nikhil Suhas Khambe
B.Tech (Bio-tech)

Note: The report refers only to submitted sample. It cannot be used for court purpose. We are not responsible for any legal matter.
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Figure 1: Pongammia Pinnata – Characterization

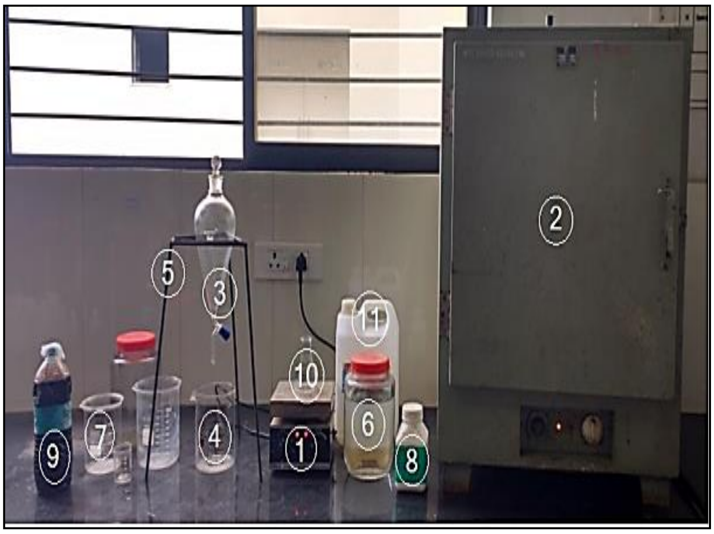

Figure 2: Biodiesel synthesis setup

Figure 2 Biodiesel synthesis setup consists of, 1-Magnetic Stirrer with Hot Plate, 2- Ovens, 3- Separating flasks, 4- Beakers, 5- Stands, 6- Ethanol, 7- PTSA (P-toluene sulphuric acid), 8- CaO (Calcium oxide), 9- Pongamia pinnata oil, 10- Conical flask, 11- Deionized

C. *Biodiesel production process*

Figure 3 shows the detailed process of biodiesel production for nonedible P. Pinnata using transesterification and heterogeneous nano catalyst PTSA and CaO with various parameters such as acidic value, moisture content, viscosity, octane number, flash, fire point and other standard parameters as per ASTM.

The production of biodiesel from *P. Pinnata* oil (Pongamia pinnata) from the above flow chart involves a series of chemical processes, including esterification, transesterification, and purification, to ensure high-quality fuel. The process begins with the extraction of *P. Pinnata* oil from the seeds of the Pongamia pinnata tree, serving as the primary raw material.

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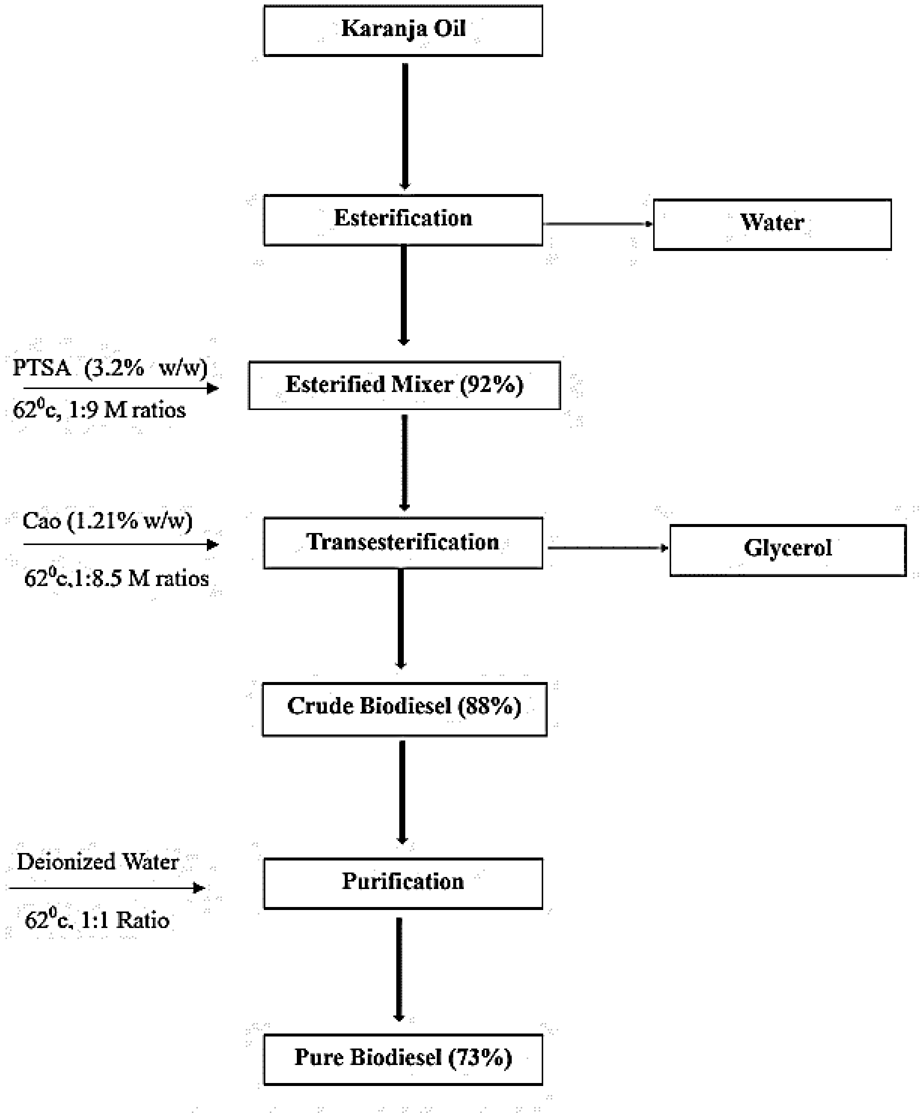

Figure 3: Flow chart of the Biodiesel production process

Due to its high free fatty acid (FFA) content, esterification is required to minimize soap formation during transesterification. In this step, the oil is reacted with methanol or ethanol in the presence of an acid catalyst (such as sulfuric or hydrochloric acid), resulting in an esterified mixture with a significantly reduced FFA content and a conversion efficiency of approximately 92%. Water is generated as a by-product. The esterified mixture then undergoes transesterification, where triglycerides are converted into biodiesel (methyl esters) and glycerol using methanol and a base catalyst like sodium hydroxide (NaOH) or potassium hydroxide (KOH). Glycerol, a by-product of this reaction, is separated from the biodiesel. The resulting crude biodiesel, with an initial purity of around 88%, contains residual impurities such as unreacted alcohol, catalyst residues, and glycerol traces.

To achieve the required fuel quality standards, the crude biodiesel undergoes purification through processes such as washing, drying, and distillation, ultimately yielding purified biodiesel with a final purity of 73%. This refined biodiesel serves as a sustainable alternative to conventional diesel, contributing to cleaner energy solutions and reducing environmental impact.

D. *Transesterification*

Following is Figure 4 which shows the transesterification chemical reaction that converts FFA nonedible P. Pinnata oil into ethyl esters i.e. biodiesel and glycerine as byproducts that can be used for other applications.

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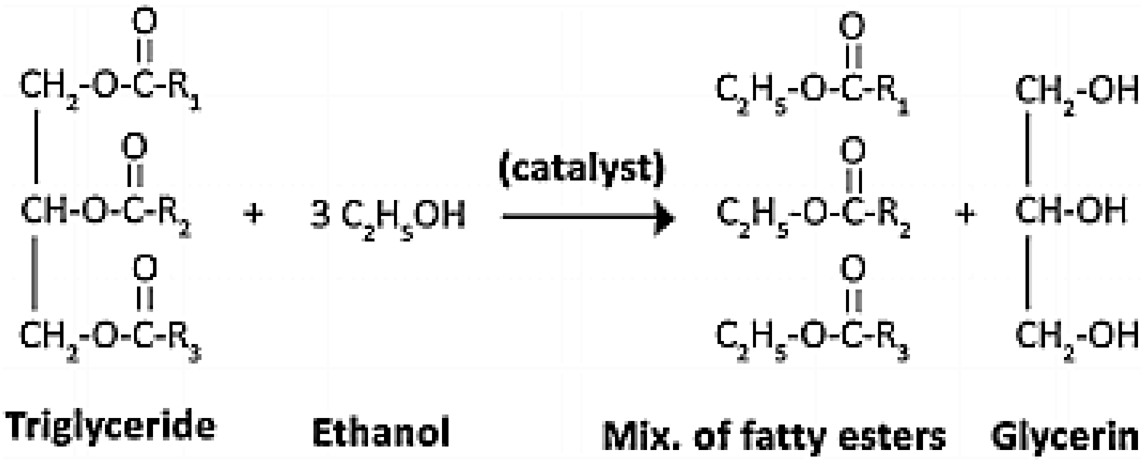

Figure 4: Transesterification chemical reaction

Transesterification using ethanol is a crucial process for biodiesel production from Karanja (*Pongamia Pinnata*), enabling the conversion of triglycerides into ethyl esters (biodiesel) and glycerol. In this reaction, *Karanja* oil is treated with ethanol in the presence of a base catalyst, typically sodium hydroxide (NaOH) or potassium hydroxide (KOH), though acid catalysts like sulfuric acid may be used if the feedstock has a high free fatty acid (FFA) content. The reaction follows an 8.5:1 ethanol-to-oil molar ratio to ensure efficient conversion. During transesterification, the ethanol is activated by the catalyst, which facilitates the breakdown of triglycerides, replacing their glycerol backbone with ethanol to form ethyl esters as the primary product and glycerol as a by-product. The reaction is highly efficient, but ethanol-based transesterification requires careful water removal to prevent saponification. After completion, phase separation occurs, with biodiesel forming the upper layer and glycerol settling at the bottom due to density differences. The crude biodiesel undergoes purification through washing and drying to remove residual ethanol, catalyst traces, and other impurities, ensuring it meets fuel quality standards. Ethanol-based transesterification offers benefits such as improved renewability and lower toxicity compared to methanol-based processes, making *P. Pinnata*-derived biodiesel a sustainable and environmentally friendly alternative to conventional diesel.

Esterification and Transesterification Method Process-


Fig a: Measuring feedstock


Fig b: Ethanolysis and esterification


Fig c: Esterification Separation

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Fig d: Transesterification

Fig e: Transesterified oil &gly

Fig f: Water-washed process

Fig g: Water-washed process

Fig f: Water-washed process for getting BD

Fig a -c is the esterification process and Fig d is transesterification, while Fig e shows Transesterified oil i.e. ethyl ester of *P. Pinnata* oil and byproduct glycerine, Fig G h shows the final water washing process that provides the final biodiesel.
After the final biodiesel is prepared with a reaction temperature of 60C and reaction time of 120 min using a catalyst and the respective molar ratio 8.5:1 the final yield is characterized and compared it properties to meet the available infrastructure and international standards.

E. *Standards for Evaluation of Biodiesel Properties*
To ensure consistent quality and compatibility of biodiesel as a fuel, various international organizations have established standards for evaluating its properties. These standards provide guidelines for testing and assessing biodiesel's characteristics. Some of the key standards include:

ASTM D6751: This standard by the American Society for Testing and Materials (ASTM) outlines specifications for biodiesel used in diesel engines. It covers parameters like kinematic viscosity, cloud point, pour point, cetane number, acid value, sulfur content, and oxidation stability.

EN 14214: The European standard EN 14214 specifies requirements for fatty acid methyl esters (FAME) as diesel fuel. It includes criteria for density, kinematic viscosity, flash point, cetane number, oxidation stability, and more.

ISO 24256: This International Organization for Standardization (ISO) standard provides guidelines for determining the cold filter plugging point (CFPP) of biodiesel and its blends. CFPP indicates the lowest temperature at which the fuel can still flow through a filter.

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EN 14112: This standard outlines the method for determining the ester content of fatty acid methyl esters, which is an essential parameter for assessing biodiesel quality.
ASTM D613: It covers the test method for measuring the cetane number of diesel fuels, including biodiesel. A higher cetane number signifies better ignition quality.
ASTM D445: This standard outlines the procedure for measuring the kinematic viscosity of biodiesel, an important property affecting fuel atomization and combustion efficiency.
ASTM D2500: It provides the test method for determining cloud point, which is the temperature at which paraffin wax crystals start to form in the fuel.
EN 15751: This standard specifies a method for measuring the cold soak filterability of biodiesel and its blends, assessing their performance in cold conditions.
ASTM D5773: It outlines the procedure for determining the cloud point and pour point of biodiesel, critical parameters for fuel operability in cold weather.
EN 14103: This standard specifies the method for determining the acid value of biodiesel, reflecting the presence of free fatty acids that can affect fuel stability.

These standards ensure that biodiesel meets quality and performance requirements, facilitating its seamless integration into existing engine systems and fuel infrastructure. Compliance with these standards promotes consumer confidence, environmental benefits, and reliable engine operation when using biodiesel as an alternative fuel source.

III. RESULT & DISCUSSION

F. *Molar ratio 7 % Yielding of Biodiesel*

The molar stands as the important parameter that takes part in the biodiesel synthesis process for any of feedstock used. The molar ratio defines the proportion of ethanol and catalyst that be used for biodiesel production. Each different feedstock defines a different molar ratio of the optimized proportion of bio-oil and ethanol to be used and the content of

Table 1: MOLAR RATIO AND % YIELD OF BIODIESEL

Temperature °C	Molar Ratio (Oil to ethanol)	Catalyst concentration (wt.%) (PTSA)	Reaction Time (min)	% Yield (Esterified Mixer)	Molar Ratio of Esterified Mixer to ethanol	Catalyst concentration (wt.%) (Cao)	% Yield (Biodiesel)
62	1:10	3.2	120	84	1:10	1.21	79
62	1:9	3.2	120	92	1:9	1.21	81
62	1:9	3.2	120	92	1:8.5	1.21	88
62	1:8	3.2	120	90	1:8	1.21	87
62	1:7	3.2	120	86	1:7	1.21	71

The maximum yield observed in biodiesel production is with a molar ratio of 1:8.5. however, with the process in appropriate room reaction and precise care at condensation and volatility of alcohol, the yield of 92% is achieved

G. *Calorific Value*

The calorific value of Karanja oil underwent meticulous characterization according to ISO/IEC 17025:2017 standards within a certified laboratory. By strictly adhering to these exacting standards, we ensure the accuracy, reliability, and traceability of the obtained calorific value data. Table 3 showcases the calorific value results of the Karanja oil biodiesel blend. This thorough analysis establishes a robust foundation for further research and development endeavors aimed at leveraging Karanja oil as a sustainable and viable resource in diverse applications, notably in biodiesel production and industrial processes.

Table 2: Calorific Value of Biodiesel Blend

Biodiesel Blend	Calorific Value (KJ/kg)
Karanja oil	3700
B50	40195.69
B20	41881.84
B10	42120.32
B5	42647.51
Diesel	43000

The above table 2 shows the B5, B10, and B20 blends of biodiesel with calorific values that are nearer to the calorific value of conventional biodiesel. So, with this biodiesel blend the available infrastructure i.e. internal combustion engine can be used without any modifications. However, emission and material compatibility can be the area of research that can reveal more about material compatibility and the meeting emission, ASTM standards.

IV. CONCLUSION

The study successfully demonstrates the production of biodiesel from Karanja oil using a two-step process involving acid-catalyzed esterification and nano-catalyst transesterification. The optimized reaction conditions resulted in high biodiesel yields of up to 92%, with a final purified yield of 73% after multi-washing. The use of nano-CaO as a catalyst proved effective in enhancing the transesterification process. The produced biodiesel, when blended with conventional diesel at 5%, 10%, and 20% ratios, aligns with industry standards and Indian biodiesel policy, offering a viable alternative to petroleum-based fuels. The findings indicate that Karanja biodiesel holds significant potential for sustainable energy applications. Future research should focus on scaling up production, evaluating long-term engine performance, and assessing environmental benefits to facilitate broader adoption of biodiesel as a renewable energy source.

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V. ABBREVIATION

FFA: Free Fatty Acid
KOEE: Karanja oil Ethyl ester
KOME: Karanja oil methyl ester
BD: Biodiesel
MR: Molar ratio
CJCO: Crude Jatropha Curcas Oil
WFOME: waste fried oil methyl ester
RSM: Response Surface Methodology
AFR: Air Fuel Mixture ration
PTSA: p-toluenesulfonic acid
CaO: Calcium Oxide
IP: Indicated power
BSFC: Brake-specific fuel
BTE: Brake thermal efficiency
P. Pinnata: Pongamia Pinnata