

Original Research

## Cost-Effective Biodiesel Synthesis from Waste Marble Powder as A Green Heterogeneous Catalyst Using Pongamia Pinnata Oil

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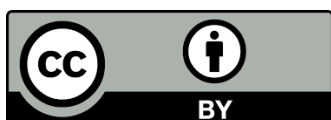
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### Abstract

Waste marble powder (WMP) is Investigated for developing heterogeneous catalysts by calcination method and used in biodiesel synthesis by esterification-transesterification process from Pongamia Pinnata oil. Hammet indicators, Fourier transform infrared (FT-IR), Thermogravimetric analysis, and X-ray powder diffraction (XRD) techniques were also studied for the characterization of developed catalyst from waste marble powder (WMP). The conversion of calcium carbonate to calcium oxide in marble powder was found at 800°C after calcination. The maximum biodiesel yield reached about 94% using a 3.5 wt% catalyst, 9:1 methanol to Pongamia pinnata oil molar ratio, and 2.5 hour process time at 65°C. The biodiesel purity was tested by gas chromatography analysis. The catalyst stability was tested by recyclability test and found a small decrease in biodiesel yield up to 5 recyclability runs. The solid heterogeneous catalyst from WMP proves that the harmful waste could be converted and used as an economically efficient solid heterogeneous catalyst for sustainable biodiesel production from Pongamia Pinnata oil.



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## Keywords

Biodiesel; transesterification; waste marble powder; pongamia pinnata oil

## 1. Introduction

Recently, green energy sources such as biomass, geothermal, solar, and biofuels have been gaining much attention worldwide because of increasing fuel prices, being environment-friendly, and boosting the economy of the country [1, 2]. In this context, Biodiesel preparation from edible and non-edible oil as a green fuel has caught the attention of researchers [3]. Biodiesel also called fatty acid methyl ester (FAME) is prepared by an esterification-transesterification process in which oil reacts with alcohol and catalyst (homogeneous or heterogeneous) at given reaction conditions. Consuming eatable oils like soybean oil, palm oil, groundnut oil, mustard oil, etc. for biodiesel preparation may be unattainable as a human meal. Therefore, the optional way to overcome the problems occurring by edible oil as biodiesel feedstock is explored. Many researchers explored non-eatable oil because it is not used for human feed and is easily obtainable [4]. To accelerate the esterification-transesterification procedure, different homogeneous and heterogeneous catalysts are used for biodiesel preparation [4]. Potassium hydroxide, sodium hydroxide, and sulphuric acid are homogeneous catalysts that yield high biodiesel with shorter reaction time [5]. However, using a homogeneous catalyst increases the pH of biodiesel, wastewater generation in the water purification step, and also very difficult of catalyst separation. So heterogeneous catalyst can be an attractive solution with many benefits such as reproducibility, easy operation procedure, simple way of catalyst, and product separation [6]. Various types of heterogeneous catalysts have been discovered for biodiesel preparation in transesterification reactions such as zeolites [7, 8], alkaline earth oxides [9, 10], zinc aluminate [11, 12], etc. The high cost and special expertise required for heterogeneous catalyst synthesis draw attention to waste used as catalytic feedstock for cost-efficient biodiesel preparation.

According to past data from Turkey, daily 12,875 tons of marble dust was generated as waste material. After dumping it on forestland roads, it increases the serious health and environmental risks [13]. Similarly other wastes such as eggshells (worldwide 16.6-41.5 million tons yearly), fly ash (83.64 million tons yearly in India), and chicken bones (worldwide 25 lakh tons yearly) were also generated in huge quantities and discarded in landfills, municipal dumps and ash ponds etc. [14-17]. Countless wastes have been tested as a catalyst like eggshells [18, 19], marble powder [20, 21], red mud [22], snail shells [23, 24], fly ash [25], and bones [26, 27]. After investigation, Calcium oxide-derived waste material is found to be one of the most efficient catalytic materials for low-cost biodiesel synthesis [28]. So here, we explored and synthesized waste marble powder as a heterogeneous catalyst for cost-effective biodiesel production. The novelty of this study is to convert the waste marble powder into an environment-friendly heterogeneous catalyst and use it in low-cost biodiesel production from Pongamia Pinnata oil. This study aimed to develop a heterogeneous catalyst, i.e., calcium oxide after calcination of waste marble powder, used in biodiesel production through a two-step transesterification reaction. Pongamia Pinnata oil was used as a feedstock in this study. Gas Chromatography (GC) analyses were done for biodiesel characterization. The recyclability test was also tested for developed heterogeneous catalysts.

## 2. Materials and Methods

### 2.1 Materials and Chemicals

A natural crushed marble powder was obtained from Jaipur, Rajasthan, India and Pongamia Pinnata oil was obtained from the manufacturer. Methanol and sulphuric acid with 99.99% purity were obtained from Apurv chemicals, Nasik, India in this study.

### 2.2 Characterization of Oil

The raw Pongamia Pinnata oil properties like kinematic viscosity, acid value, density, etc., are measured manually as described in the literature [29] as illustrated in Table 1.

**Table 1** Physical properties of Pongamia Pinnata oil.

Properties of Pongamia Pinnata oil	Unit	Value
Acid value	mg of potassium hydroxide/gm of oil	8.34
FFA	-	4.17
Density	gm/ml	0.90
Kinematic viscosity at 40°C	Cst	34.33
Saponification value	mg of potassium hydroxide/gm of oil	189.00
Molecule weight	gm/mol	892.70
Ester value	mg	180.66
glycerol	%	9.87

### 2.3 Preparation of Catalyst

The marble powder was first cleaned with tap water 2-3 times to clean off dirt and then dried in a heating oven at 105°C for 1-2 hours. The white marble powder was crushed in a pestle and mortar and calcined at 800°C for 2 hours in a muffle oven. The calcinated marble powder was cooled at ambient temperature and reused as a solid catalyst for biodiesel synthesis.

### 2.4 Catalyst Characterization

To analyze the powder diffraction pattern, X-ray Powder Diffraction (XRD) was used for the developed catalyst sample. The infrared spectrum was recorded on an FTIR spectrometer (Perkin Elmer spectra two) in the 400–4000  $\text{cm}^{-1}$ . The KBr pellet technique prepared FTIR sample. Thermal gravimetric analysis (TGA) tested the prepared catalyst's thermal stability. TGA was used by a thermal analyzer at 27°C-900°C in an inert nitrogen atmosphere with a heating rate of 20°C  $\text{min}^{-1}$ . The basicity of the sample was tested by the Hammett indicator method.

### 2.5 Esterification-Transesterification Reaction Method

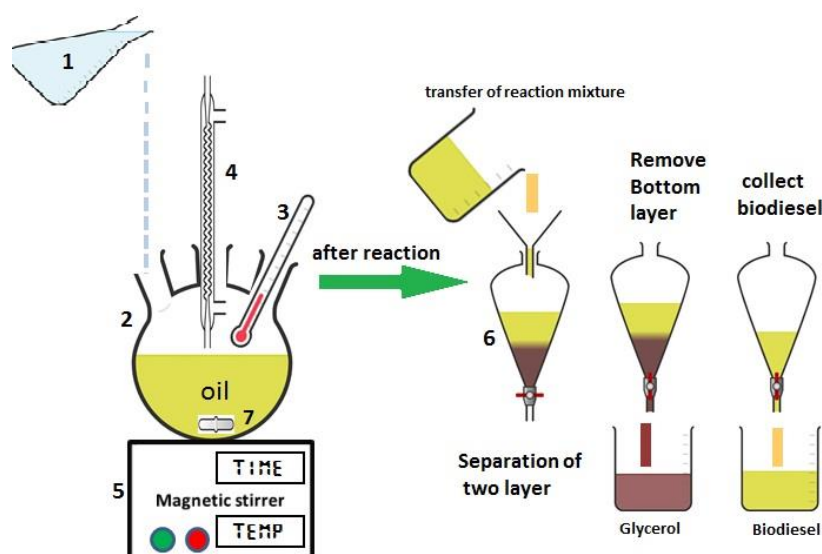
In the initial step, the esterification reaction was chosen and performed to reduce acid value at 65-70°C by sulphuric acid as a homogeneous acid catalyst. The reaction was done in three necks round bottom flask of 500 ml capacity. The middle neck was attached with a reflux condenser, one side neck was connected with a temperature controller, and the other neck was used for feeding

the samples in the flask. The whole set-up was put on the magnetic stirrer with a hot plate as illustrated in Figure 1. A total of 100 ml of Pongamia Pinnata oil was preheated (60°C) on a hot plate and then add 1-2 ml acid catalyst i.e. H<sub>2</sub>SO<sub>4</sub>, and methanol/ oil in a 6:1 molar ratio in the heated oil. Two hours of reaction time were fixed to ensure a complete esterification reaction under continuous stirring. After two hours, the esterification reaction mixture was cool down at normal temperature and keep in the separatory funnel for the separation of phases. Two discrete phases appeared. The upper phase consists of the esterified sample which was separated and stored in another beaker for the next process. The bottom part of the separatory funnel consists of glycerol.

In the last step, a Similar experimental setup was used as described above. Typically, a 3.5 wt% catalyst sample was suspended in methanol in a flask under continuous stirring for 30-40 minutes. Subsequently, A total of 100 ml heated esterified Pongamia Pinnata oil was added into the above methanol catalyst solution for 2.5 hours at 65°C. The methanol/oil molar ratio was chosen as 9:1 in this research. After 2.5 hours, the transesterification reaction mixture was cool down at normal temperature and keep in the separatory funnel for the separation of phases. Two discrete phases appeared. The upper phase consists of the product sample (biodiesel) which was separated and stored in any other beaker for the analysis as illustrated in Figure 1. The bottom part of the separatory funnel consists of glycerol and a solid catalyst. A centrifuge separator separated the catalyst from glycerol. The biodiesel yield was calculated as given in the following equation 1:

$$\text{Biodiesel yield}(\%) = \frac{\text{weight of biodiesel}}{\text{weight of esterified Pongamia Pinnata oil}} \times 100 \quad (1)$$

The catalytic recyclability of the catalyst was also tested by repeating experiments of the transesterification reaction. The catalyst was centrifuged, separated, and dried from the mixture, and then used for the next run. The reaction conditions were used in each run : catalyst amount 3.5 wt%; methanol/oil molar ratio: 9:1; reaction temperature: 65°C, reaction time 2.5 hours.



**Figure 1** Transesterification reaction procedure for biodiesel production. 1. Methanol & catalyst mixture 2. 3-neck round bottom flask 3. Temperature-sensor 4. Condenser 5. magnetic stirrer with hot plate 6. Separating funnel for separation of biodiesel & glycerol 7. Magnetic stirrer bar.

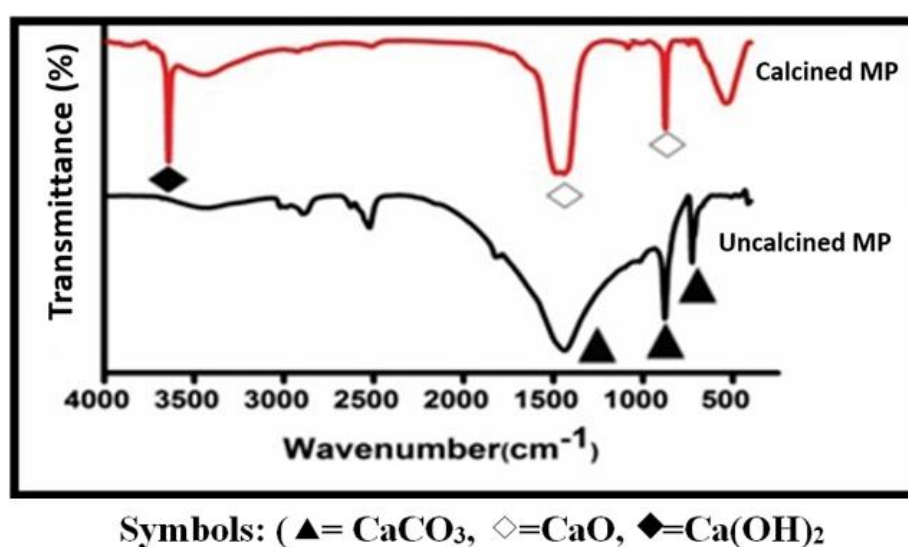
## 2.6 Biodiesel Characterization

The purity of the methyl ester layer was estimated using a gas chromatograph (Dani Master GC Instruments, Italy). The reaction conditions are as follows: capillary column length of 30 m, injector temperature: 240°C, detector temperature: 280°C and column heating flow rate: 40°C min<sup>-1</sup> for 100°C-200°C, 2°C min<sup>-1</sup> for 200°C-232°C, and 30°C min<sup>-1</sup> for 232°C-280°C. Initially, a 100 mg standard mixture of pure methyl esters (C<sub>8</sub>–C<sub>24</sub>) was purchased from Sigma Aldrich. Then 2 ml of hexane was dissolved in 0.5–1 ml of standard solution and injected through the syringe into the capillary column of GC. The resulting graph showed the elution time of individual biodiesel components used for prepared biodiesel analysis.

## 3. Results and Discussion

### 3.1 FTIR Spectroscopy Analysis

FTIR spectroscopy analysis of marble powder (after calcination and before calcination) was used to obtain FTIR spectra as illustrated in Figure 4. Two major adsorption bands were found at 875 and 1433 cm<sup>-1</sup> which shows the presence of CaCO<sub>3</sub> as illustrated in Figure 2 [30,31]. The intensity of these absorbance bands decreases because of losing the carbonate group and formation of CaO and Ca(OH)<sub>2</sub> during calcinations at 800°C. One sharp band of OH group attached with calcium atoms appears at 3642 cm<sup>-1</sup>. as illustrated in Figure 2 [29, 30].



**Figure 2** FTIR spectrometer analysis of heterogeneous catalyst. Symbols: (▲ = CaCO<sub>3</sub>, ◇ = CaO, ◆ = Ca(OH)<sub>2</sub>).

### 3.2 XRD Analysis

To investigate the crystalline structure of the catalyst, the X-ray diffraction analysis was performed as illustrated in Figure 3. Figure 3 shows the XRD graph of marble powder (MP) and calcined Marble powder (CMP). The presence of calcium carbonate in the catalyst showed major diffraction peaks at 29.37°C, 30.90°C, and 41.05°C with some other impurities present in marble powder like Silica di oxide, magnesium hydroxide etc. After calcination, new crystalline phases

appear due to CaO at 33.90°C, 37.12°C, and 42.67°C in calcine MP as described in the literature [31, 32]. It proves calcium carbonate is completely converted into calcium oxide as a heterogeneous catalyst.

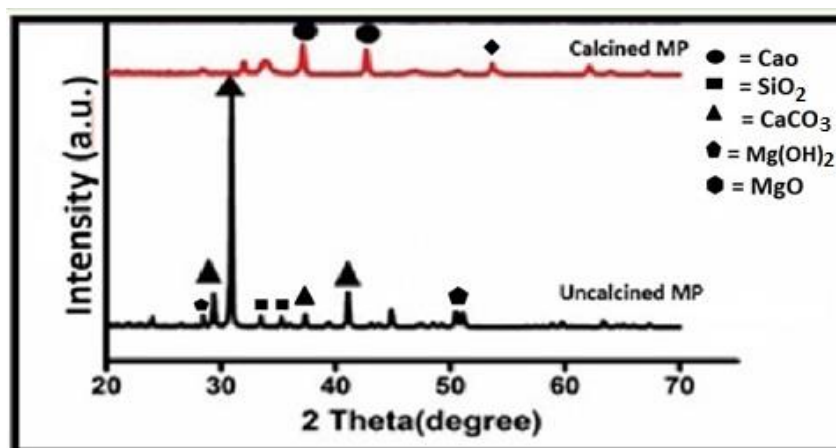


Figure 3 XRD analysis of heterogeneous catalyst.

### 3.3 Hammett Basicity Method

The Hammett *basicity* indicator method was chosen for the basicity testing of solid catalysts. A 5 ml of methanol was mixed with 50 mg calcined marble powder under shaking and add 2-4 drops of Hammett basicity indicator. The above solution was left for 2-3 hours for equilibration. If the solution color changes, the indicator basicity is weaker than the basicity of a solid catalyst. If the solution shows no color change, the indicator basicity is stronger than the basicity of the solid catalyst [33]. The basic strength of the uncalcined and calcined marble powder was tested according to color changes as illustrated in Table 2. Hammett basicity indicators used in this study as per basic strength are as follows: phenolphthalein (Base strength (H<sub>-</sub>) = 9.8), indigo carmine (Base strength (H<sub>-</sub>) = 12.2), 2,4 dinitro aniline (Base strength (H<sub>-</sub>) = 15.0) and 4-chloride-2-nitroaniline (Base strength (H<sub>-</sub>) = 17.2). After observation, pure calcined marble powder showed higher base strength in the 9.8 < H<sub>-</sub> < 17.2 than the base strength of uncalcined marble powder.

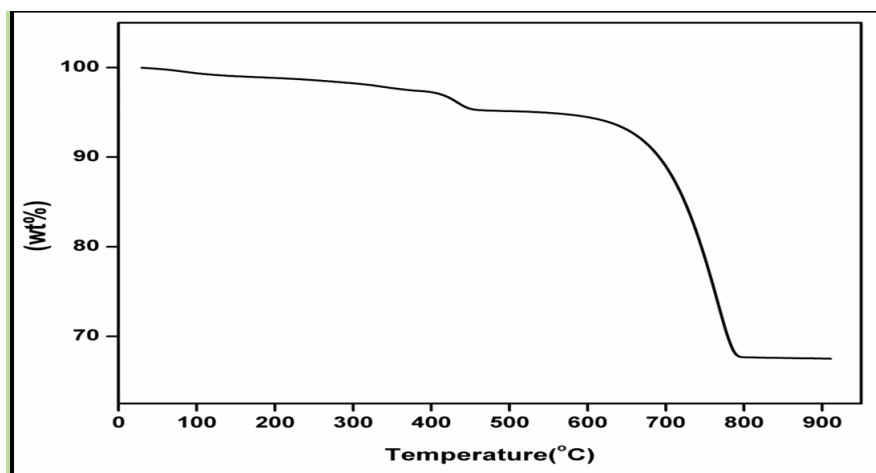
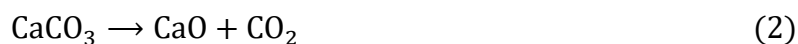
Table 2 Measured the basicity of developed solid catalysts.

Catalyst	Original color	Basic color	Basic strength (H <sub>-</sub> )
Waste Marble Powder (WMP)	white	Reddish-orange	9.8-12.2
Calcined Marble powder (CMP)	white	orange	9.8-17.2

### 3.4 Thermal Gravimetric Analysis (TGA)

Figure 4 depicts TGA curves of pure marble powder before calcination. The uncalcined marble powder indicated two weight losses in the temperature range of 367.22°C-499.98°C and 509°C-700.3°C respectively. The first decrease in wt% is seen due to the separation of Ca(OH)<sub>2</sub> in the range 367.22°C-499.98°C. The second decrease in wt% is seen due to the chemical reaction in which CO<sub>2</sub> is released from the CaCO<sub>3</sub> and only CaO is found to be the main component in catalytic material.

The second decrease in wt% is seen in the temperature range of 509°C-700.3°C [31-33]. The thermal stability of the marble powder suggests the best calcination temperature is 700°C-800°C.



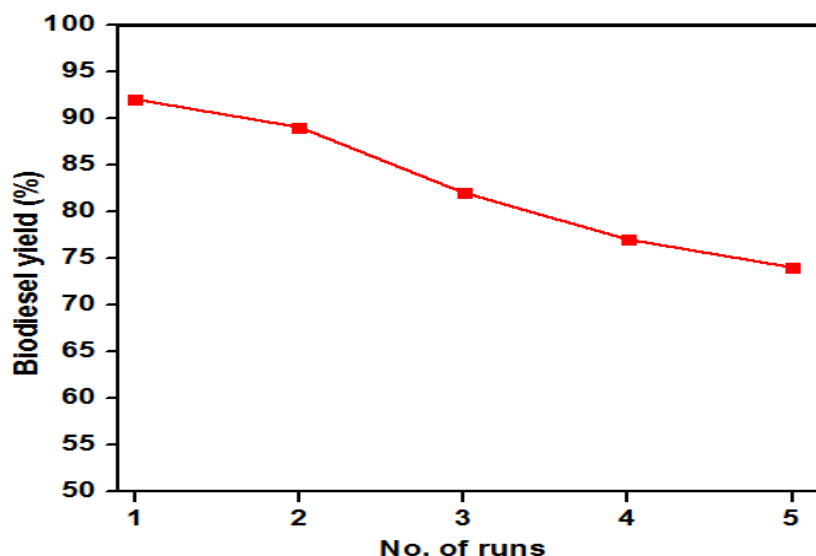
**Figure 4** Thermogravimetric Analysis of marble powder before calcination.

### 3.5 Recyclability Test

Catalyst recycling is very important for the environment and economic sense. As illustrated in Figure 5, the transesterification reaction was performed at the same reaction conditions with the same experimental setup as used in this study for testing the recyclability efficiency of the calcined marble powder in biodiesel production.

After the transesterification reaction, the biodiesel and glycerol layers were separated using a separating funnel, and then the solid catalyst was separated from glycerol using a centrifuge. The separated catalyst was again used for another transesterification cycle. The catalytic activity of calcined marble powder reduced from 94% to 73% within 5 runs as illustrated in Figure 5. The calcium leaching of the calcium oxide catalyst plays a major role for decreasing catalytic activity and biodiesel yield in recyclability transesterification reaction test. CaO leaching occurs due to vigorous mixing of the reactant with catalyst in the reaction medium. From literature it was found that calcium oxide reacts with glycerol (by product in transesterification reaction) gives diglyceroxide which is highly soluble in methanol and results highly Ca leached catalyst [34, 35]. After 5 runs, no recyclability test was performed because the catalyst was crushed into small particles due to strong stirring in the transesterification process.

This results in a loss of catalytic activity.



**Figure 5** Recyclability test of solid catalyst.

Table 3 showed the comparable study of different waste-derived solid catalyst for biodiesel production based on feedstock, operating conditions, biodiesel yield and catalyst recyclability. From this table, it can be justify that we observed better yield i.e. 94% under optimum reaction conditions with good biodiesel properties.

**Table 3** Comparative study of heterogeneous catalysts derived from waste for transesterification reaction.

Catalyst used	Feedstock	Operating Conditions				Recyclability	Yield (%)	References
		Temp (°C)	Alcohol to oil molar ratio	Time (hour)	Catalyst concentration (wt%)			
Marble dust	Canola oil	65	1:6	3	3	Upto 3 cycles	89.3	[36]
Waste shell	Mustard oil	65	12:1	3	7	Upto 4 cycle	93.3	[37]
Ostrich bone	Waste cooking oil	60	15:1	4	5	-	90.5	[38]
Abalone shell	Palm oil	65	12:1	3	7	Upto 5 cycle	87.5	[39]
Scallop shell	Rapeseed oil	60	12:1	4	9	Upto 4 cycle	83	[40]
Waste animal bone	Waste frying oil	70	12:1	4	10	Upto 10 cycle	96% FFA conversion	[41]
Marble powder	Pongamia Pinnata oil	65	9:1	2.5	3.5	Upto 5 cycle	94	Current research



### 3.6 Biodiesel Properties Measurement

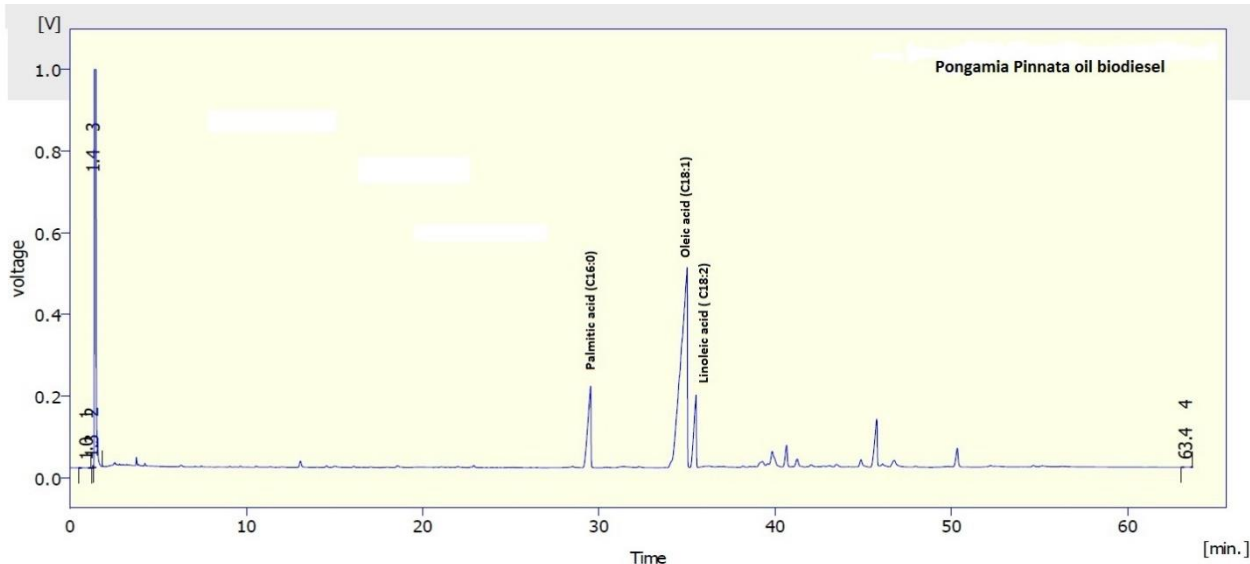
The Pongamia Pinnata oil biodiesel properties were tested manually and compared with ASTM D6751 Standard as illustrated in Table 4 [42].

**Table 4** Measured properties of Pongamia Pinnata oil biodiesel.

Properties	Unit	Biodiesel	ASTM Standard
Density	(gm/ml)	0.89	0.860-0.900
Kinematic viscosity	at 40°C (Cst)	4.83	1.9-6.0
Acid value	(mg of KOH/gm of oil)	0.47	0.50
Cloud point	°C	6	-3 to 12
Pour point	°C	-3	-15 to 10
Flash point	°C	159	130-170
Fire point	°C	178	140-215

#### 3.6.1 GC Testing

The graph of Pongamia Pinnata oil biodiesel shows fatty acid methyl ester components such as linoleic acid, oleic acid, and palmitic acid as illustrated in Figure 6. GC testing compared with the standard graph and confirmed the purity of fatty acid methyl ester from Pongamia Pinnata oil [43].



**Figure 6** Gas Chromatography analysis of produced biodiesel.

## 4. Conclusions

A good yield was found after converting Pongamia Pinnata oil to biodiesel using waste marble powder as a heterogeneous catalyst. Different analytical techniques such as FTIR, XRD, TGA, and GC are used for catalyst and biodiesel characterization, respectively showing the strong basicity of the developed catalyst for biodiesel synthesis. This catalyst shows good biodiesel yield i.e. 94% at the reaction conditions such as reaction time of 2.5 hours, methanol to oil molar ratio of 9:1, reaction

temperature of 65°C, and catalyst loading of 3.5 wt%. The developed catalyst from marble powder exhibits excellent catalytic activity and stability and cost-effective biodiesel production.

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### **Author Contributions**

Dr. Jharna Gupta has collected the data and written the manuscript. Dr. Madhu Agarwal has given valuable suggestions for finalizing the manuscript.

### **Competing Interests**

The authors declare that they have no conflict of interest regarding this publication.

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