Experimental Optimization of Biodiesel Synthesis from Nonedible Feedstock *Ceiba Penandra* Using Nanocatalyst

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Abstract

The demand for petroleum-based fuels surged due to rapid industrialization and population growth, which raised prices and had a negative impact on the environment. In order to produce biodiesel on a wide scale, which is a sustainable, long-lasting alternative, it needs readily accessible feedstocks. In particular, Ceiba penandra is abundantly available in non-edible plant in Southern India. An effective heterogeneous nano-catalyst made of CaO was prepared, evaluated using FTIR, XRD, SEM, and EDX to determine its physical and chemical characteristics, and used for both transesterification and esterification simultaneously. Ceiba penandra's biodiesel yield was achieved by over 95% by the modification of key reaction parameters. The optimal values were for the methanol oil ratio (0.32 v/v), catalyst usage (5 wt.%), mixing intensity (750 rpm), mixing time (80 min) with the constant temperature of 70°C. In order to prove its compatibility in the IC engine, the ASTM and EN guidelines and a chromatograph were used to characterise the fatty acid profile and verify its qualities.

Keywords: *Ceiba penandra oil, nanocatalyst, transesterification, optimization, biodiesel*

Introduction

Demand for fossil fuels is increased by the widespread use of these fuels in the manufacturing, automotive, and agricultural sectors. People currently have to deal with the fluctuating cost of petroleum fuels as well as air pollution from vehicle exhaust. Global warming will result if this scenario is maintained because more greenhouse gas emissions will be released into the atmosphere. As a result of global warming, sea levels are rising and many living species perish. Switch to a replacement that should be renewable and sustainable in light of the increasing demand for energy and environmental concerns [1]. According to the International Energy Agency (IEA), biofuel is a future sustainable and renewable energy source in comparison

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to other energy sources like wind, solar, and hydropower. Modern studies have discovered that biodiesel made from numerous renewable sources is beneficial for lowering greenhouse gas emissions in the atmosphere as well as the demand for petroleum fuel [2, 3]. For the past 20 years, biodiesel has been praised for its environmental friendliness, sustainability, and biodegradability. It may be used in a diesel engine directly, without needing to be modified, and has a high calorific value, cetene number, flash point, and low sulphur content [4].

The first time biodiesel was made; there was a food vs. fuel dilemma and deforestation because it was made from edible oils like rapeseed oil, coconut oil, and sunflower oil. Non-edible oils are utilised to prevent these problems [5]. There are many non-edible plants to choose from in India. *Ceiba penandra* is a tropical non-edible plant that is native to Southeast Asia, is grown in India, Sri Lanka, and tropical America, and is typically found on wastelands. It is drought tolerant and typically grows in humid and subhumid tropical regions. It has ellipsoid-shaped pots and grows to be 60 to 70 m tall, 10 to 25 cm length, and 3 to 6 cm in diameter. It has 17% fibre, which is primarily utilised in pillows and mattresses, while the seeds make up around 25–30 wt% of each pod and may produce 1280 kg/ha of oil annually on average [6].

In a series of chemical reactions known as transesterification, triglycerides from the oil are combined with shorter-chain alcohols while a catalyst is present. In this process, the catalyst is essential for effectively accelerating the process [7]. Commercially, the transesterification process uses homogeneous catalysts such KOH, NaOH, and CH_3OK. The quality and amount of biodiesel are impacted by homogeneous catalysts because they are powerful catalysts that rapidly cause soap production and cannot be separated after the process. Heterogeneous catalysts are now preferred because of their capacity to be recycled, cost effectiveness, and environmental safety. Additionally, it is a weak catalyst that takes time to produce soap and renders the reaction irreversible [8].

Low cost materials and production techniques are also greatly valued for the manufacture of commercially viable biofuels. Resources that were naturally occurring and close by would have produced respectable results, which would have reduced the eventual cost of the biofuel. Additionally, the chemicals used in the conversion process are crucial in determining the final cost of the biofuel. Natural resources will be the most cost-effective for the selection of raw materials as well as other elements because the initial investment and production costs are a significant factor in the economic evaluation [9, 10].

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Due to its better conversion efficiency and shorter conversion time, CaO-based nanocatalysts are currently becoming more and more popular in the manufacture of biodiesel. The majority of CaO comes from seashells, and there are still a lot of them across the world that aren't being properly used. CaO catalyst still has several limitations, such as poor activity and a slower reaction rate. CaO can be combined with an additional solid-based, alkaline nanocatalyst, such as metal oxides, hydrotalcites, or anion exchange resin, to produce biodiesel more effectively and with a greater yield [11, 12].

The primary goal of the work is to create biodiesel from second-generation Ceiba penandra feedstock, which is inexpensive and readily available locally. To increase the biodiesel's efficiency, TiO_2 Nanoparticles were added to a heterogeneous natural seashell powder to prevent the need for pretreatment (esterification) procedures. Numerous spectroscopic techniques are used to characterize the green catalyst and to prove its viability. Additionally, the response surface methodology is used to optimize the conversion process parameters, including the methanol/oil ratio, catalyst concentration, stirring speed, and processing time. Gas chromatography is used to characterize the final product, methyl ester, and to study its physiochemical characteristics in accordance with ASTM and EN standards.

Materials and methods

Various locations in Tamilnadu, India, were used to collect Ceiba penandra seeds, and an expeller machine was used to extract the oil. To determine the basic properties, the extracted oil was calculated and characterized using the right techniques. Associated Laboratory Co., Nagercoil, India provided 99% pure chemicals such as methanol, ethanol, sulphuric acid, and phenothaline indicator. For the purpose of filtering the raw oil and biodiesel to get rid of the dust, Whatman filter sheets were bought from affiliated laboratory Co. in Nagercoil, India.

High acid value oils typically require a multi-step esterification method to lower them in order to prevent soap production during the transesterification process. Due to its high catalytic activity, water tolerance, and stability due to the abundance of base and acid active sites, heterogeneous catalysts based on metal oxides may effectively execute both the esterification and transesterification processes concurrently [13]. Additionally, they are not affected by the presence of FFA, do not require neutralization, and are simple to remove from the reaction media. In order to eliminate the moisture, the oils were heated at 105°C for 30 min.

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The free fatty acid content (FFA), triglycerides, unsaponified fats, and moisture content of the oil was assessed using conventional titrimetric procedures. Free fatty acid content was measured using titration with 0.1 N KOH, and unsaponified matter was measured through titration with 0.02 N NaOH solution in alcoholic medium using Phenolphthalein indicator. Enzymatic assay was used to evaluate the triglycerides, and weight reduction was used to estimate the moisture content. The Cleaveland open cup apparatus was used to determine the flash point and fire point. A hydrometer from Mumbai-based LEIMCO was used to measure the density of the oil and the biodiesel.

Experimental setup

The Ceiba Penentra oil maintains a 1000 ml flat-bottom flask with a reflux condenser as part of its biodiesel conversion process system (transesterification). The flask with a flat bottom is set atop a hot plate magnetic stirrer. In the digital display, stirring rate and temperature may be seen. In the presence of a heterogeneous catalyst, the warmed oil was heated to a temperature of 70 °C over a period of time while being stirred smoothly. Vegetable oils contain glycerol as well as long-chain saturated and unsaturated carboxylic acids as triglycerides. The transesterification procedure converts triglycerides into glycerol and volatile esters, sometimes known as biodiesel, by sequentially reacting them with a shot-chain alcohol in the presence of a catalyst. By using a distillation process, the pure biodiesel is separated, cleaned in hot water, and then heated to drive off the moisture. The transesterification process parameters were adjusted to maximise biodiesel yield while utilizing the least amount of energy possible.

Catalyst characterization

The calcinated CaO was examined using FTIR, XRD, and SEM with EDX in order to create biodiesel. The catalyst CaO.TiO2 was found to improve biodiesel conversion. The catalyst's functional group (chemical bond) was identified using Fourier transform infrared spectroscopy (FTIR) analysis with Perkin Elmer Spectrum ASCII PEDS 4. Using a scanning electron microscope, the synthesised catalyst's shape and structure were once more investigated (SEM). Using a BRUKER ECO D8 ADVANCE diffactometer with Cu K radiation and the detector LynxEye, X-Ray diffraction (XRD) was performed to determine the catalyst's

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crystalline structure. To determine the chemical composition of the samples, the surface morphologies and particle sizes of the doped catalyst were assessed by SEM with Energy Dispersive analysis recorded by ZEISS with a BRUKER EDAX detector.

Biodiesel characterization

The fatty acid methyl ester composition of Ceiba penandra oil is characterized using Gas chromatography and mass Spectroscopy. Agilent technologies (USA) 5977 series is equipped with high performance MS capillary column (5181-8836 Agilent) with a length of 25 m and the internal diameter of 320 µm. Ultrapure (99.999%) helium is used as the carrier gas flow through the column at room temperature from 15 to 30 min to remove the air. Increase the temperature at a rate of 10 to 15 ºC/min and hold the temperature for 30 min. When the GC is ready, the oven temperature is kept at 30°C. After that, it is raised to 260°C. The GC/MSD interface should be operated between 250°C and 350°C. By looking at the percentage of peak areas with retention duration, fatty acid signature can be calculated. Engine performance is significantly influenced by density, a crucial characteristic of biodiesel. Other fuel attributes including kinematic viscosity, cetane number, iodine value, flask point, and acid value are calculated using the required processes and compared to the American and European biodiesel standards.

Optimization design

Utilizing Design Experts software version 7.0, a central composite design (CCD) based on response surface methodology (RSM) was used to improve the process parameters of the biodiesel conversion process [14]. To maximise the ester yield at the proper temperature, key process variables such methanol, catalyst, speed, and duration are changed. The ranges and levels of the process parameters are fixed in accordance with prior research and pilot tests to obtain the optimal yield, and the related experimental design is given in Table 1, which contains 30 sets of trials.

Table 1. Ranges and levels of the process parameters for transesterification process

Name of the oil	Operating parameters	Ranges and levels		
		Low (-1)	Medium (0)	High (+1)
Ceiba Penentra	Methanol:oil (v:v)	0.1	0.3	0.5

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oil	Nanocatalyst (vol.%)	2	6	10
	Stirring speed (rpm)	600	800	1000
	Time (min)	70	85	100

Results and discussion

The Ceiba Penentra seeds produced 40.2% oil when put through a mechanical oil expeller. The extracted oil was then filtered, heated, and described, and the parameters were then stated in Table 2. The phases in the esterification process, followed by the transesterification process, for the conversion of biodiesel are determined by the amount of free fatty acids (FFA), based on the employment of a catalyst.

Table 2. Properties of *Ceiba penandra* raw oil

Properties	Unit	*Ceiba penandra* oil
Acid value	(mg KOH/g)	32.12
Density	(kg/m^3)	920
Kinematic viscosity	(mm^2/Sec)	26.24
Flash point	(°C)	254
Fire point	(°C)	269
FFA	(%)	16.06
Triglycerides	(%)	81.97
Unsaponified matter	(%)	1.92
Moisture	(%)	0.05
Saponification value	-	202
Iodine value	-	71.5
Calorific value	(MJ/kg)	27.52

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FTIR (Fourier-Transform Infrared Spectroscopy) for catalyst

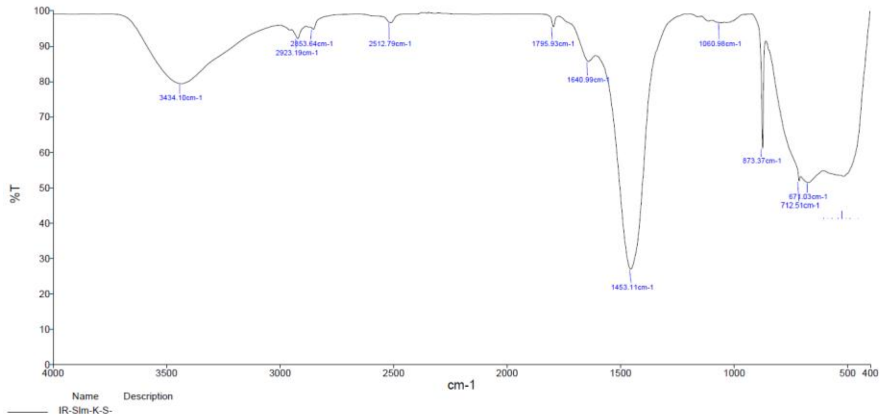

Fig. 1. FTIR analysis of the nano catalyst

The synthesised sample's FT-IR spectrum displayed a number of distinctive peaks, as shown in Figure 1. In this spectrum, the stretching vibration of O-H at 3434 cm-1 is related to the absorption band, which represents the water as moisture, and the bending vibration of O-H at 1640 cm-1. The Ti-O stretching band, which is the distinctive peak of TiO2, is credited with producing the intense peak at 671 cm-1. The vibration of the Ca-O bond is represented by the peak in the 500–580 cm-1 range.

XRD (X-ray powder Diffraction) for catalyst

The produced nano catalyst's XRD spectra are displayed in figure 2. The outcome unmistakably demonstrates that the sample contained $CaCO_3$ and TiO_2. The TiO_2 in the sample is anatase in nature. The $CaCO_3$ spectrum corresponds to the normal $CaCO_3$ JCPDS pattern of 05-0586. The sample's TiO_2 is compatible with the JCPDS spectra of anatase TiO_2 21-1272. The average crystallite size of the TiO_2 nanoparticles was 23.5 nm. The $CaCO_3$ nanoparticles had an average crystallite size of 20.6 nm. The pattern's outcome demonstrates that CaO production on TiO_2 is indicated and confirmed by the concentrations of the values.

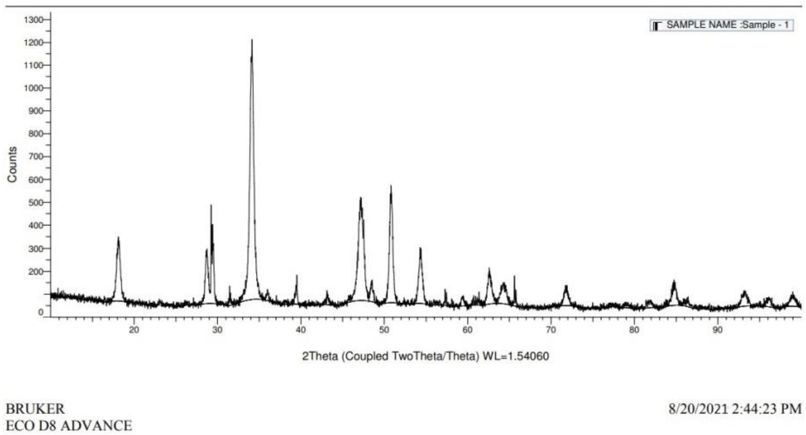

Fig. 2. XRD analysis curve of the nano catalyst

SEM with EDX (Scanning electron microscope with Energy Dispersive X-Ray Analysis)

Figures 3a and 3b display the related EDX graph and the SEM pictures of the TiO2 doped CaO nano catalyst at a magnification of 5.00kx. A superior chemical response is provided by the particle's spherical outer surface, which ranges in size from 1 nm to 200 nm. The pinnacles labelled with Ca may be seen on the range in Fig. 3c, confirming the existence of calcium oxide on titanium dioxide's outer layer.

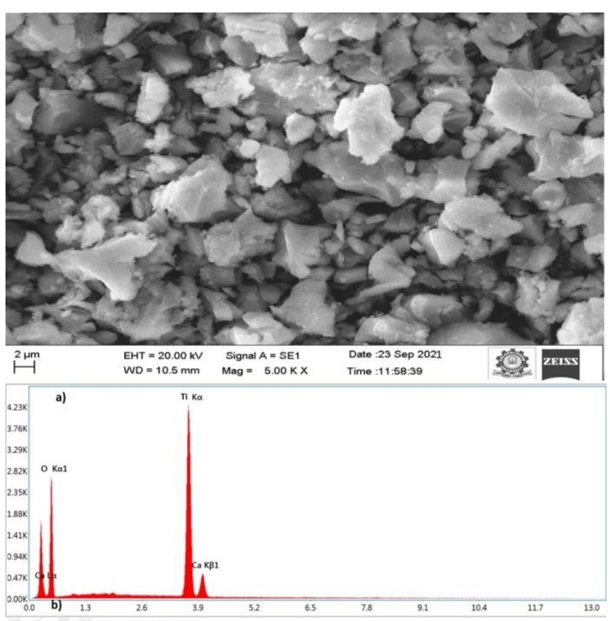

Fig. 3. SEM images of the nano catalyst at magnifications a) 50 KX and b) c) corresponding EDX graph.

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Optimization of transesterification process

Triglycerides are transformed into esters during the transesterification process in the presence of an alkali catalyst. By maximising key process variables such the methanol/oil ratio, catalyst concentration, processing time, and stirring rate, the main goal of this procedure is to maximise the ester yield. Table 3 listed the experimental strategy for ester yield along with the variables. CaO catalyst was used to produce about 80% of the biodiesel output, whereas CaO doped Al_2O_3 produced a significantly higher yield. As a result, CaO doped TiO_2 was created and used in the transesterification process with positive results; as a result, these experimental values were taken into account for improving the process parameters.

Table 3. Biodiesel yield in the transesterification process for *Ceiba penandra* nanocatalyst

Run mixed	A:Methanol:Oil (v:v)	B:catalyst (wt.%)	C:time (min)	D:speed (rpm)	yield (%)
1	0.5	10	600	70	76.2
2	0.5	2	600	100	67.3
3	0.1	2	600	70	18.2
4	0.1	2	600	100	22.1
5	0.3	6	400	85	80.2
6	0.1	10	1000	100	38.6
7	0.5	10	1000	100	91.6
8	0.1	10	600	70	26.8
9	0.5	10	600	100	89.2
10	0.3	14	800	85	82.9
11	0.1	2	1000	100	27.1
12	0.5	10	1000	70	81.4
13	0.3	6	800	85	93.3
14	0.1	6	800	85	0
15	0.3	6	800	85	93.3
16	0.3	6	1200	85	84.7
17	0.5	2	600	70	62.1
18	0.1	2	1000	70	22.4
19	0.3	6	800	85	93.3
20	0.3	6	800	85	93.3
21	0.1	10	600	100	31.2
22	0.3	0	800	85	20.7
23	0.5	2	1000	70	66.8

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24	0.3	6	800	85	93.3
25	0.1	10	1000	70	30.2
26	0.3	6	800	85	93.3
27	0.5	2	1000	100	71.5
28	0.7	6	800	85	93.5
29	0.3	6	800	55	68.7
30	0.3	6	800	115	93.5

To develop appropriate empirical models, the results were thoroughly examined. The superiority of the model is proven by the coefficient of determination ($R^2 = 0.9636$) and adjusted coefficient of determination ($R\text{-}adj^2 = 0.9297$). The anticipated model displays a comparison between the expected and actual percentages of fatty acids (Figure 4). All of the actual values are reported to be close to the anticipated values, while just a few numbers deviate somewhat from the common path. Equation (1) displays the yield percentage that the *Ceiba penandra* oil model predicts.

$$Yield = -12.74248 + 30.48247A + 0.68834B + 0.010117C + 0.20338D + 0.038191AB - 1.48932E\text{-}003AC - 3.42147E\text{-}003AD - 1.18265E\text{-}005BC + 9.12923E\text{-}004BD + 2.90032E\text{-}006CD - 32.24262A^2 - 0.049473B^2 - 5.71398E\text{-}006C^2 - 1.12727E\text{-}003D^2 \quad (1)$$

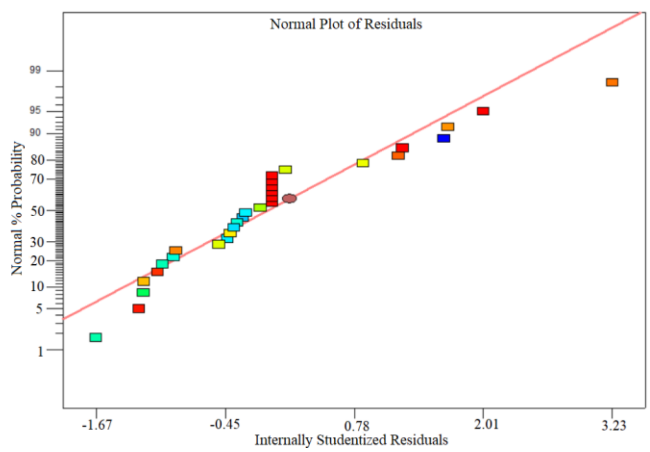

Fig. 4. Predicted Vs. actual plot attained in the transesterification for *Ceiba penandra* oil

To ascertain the statistical significance of the outcomes displayed in table 4, the mathematical regression model was assessed by ANNOVA. The experimental model was significant and the p-value for the 95% guaranteed confidence level was less than 0.05. At a 95% confidence level, ANNOVA independently assessed each empirical model using its F value and p-value [15]. Table 4 demonstrates that, with the exception of the processing time, practically all of the results for Ceiba penandra oil were statistically significant.

Table 4. Analysis of variance (ANOVA) effects for Ceiba penandra biodiesel yield for RSM model.

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	25504.81	14	1821.77	17.73	< 0.0001
A-methanol/oil	13848.01	1	13848.01	134.75	< 0.0001
B-catalyst	2244.6	1	2244.6	21.84	0.0003
C-Stirring speed	86.26	1	86.26	0.84	0.3741
D-time	451.53	1	451.53	4.39	0.0534
Residual	1541.54	15	102.77		
Lack of Fit	1541.54	10	154.15		
Pure Error	0	5	0		
Cor Total	27046.35	29			

By adjusting a single parameter throughout its range while holding the other parameters constant at the midway, the perturbation plot conveys the process parameters that are impacting the biodiesel conversion process for a greater biodiesel yield in a defined space. The Methanol/Oil Ratio is the most important factor in the optimization process, as it influences both the amount of methanol and the output of biodiesel, as shown by the deep curve A-A in Fig. 5. The remaining characteristics don't have much of an impact on the transesterification process, but they all have a significant part in the conversion process, which is covered in more detail below.

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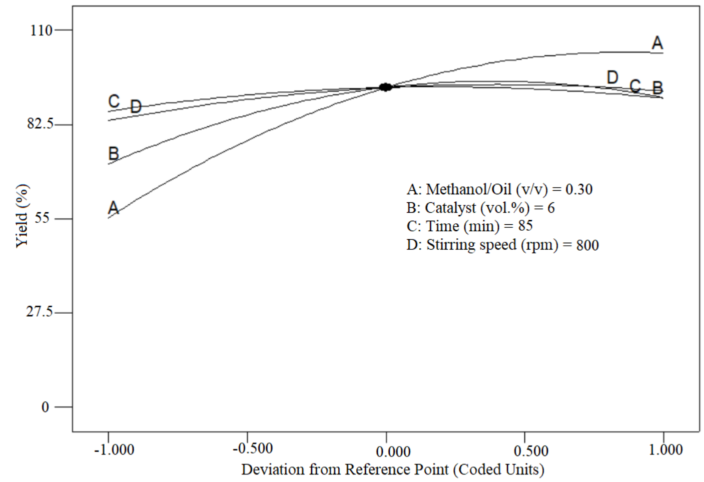

Fig. 5. Perturbation plot of the transesterification process to find the influences of the parameters for Ceiba penandra oil

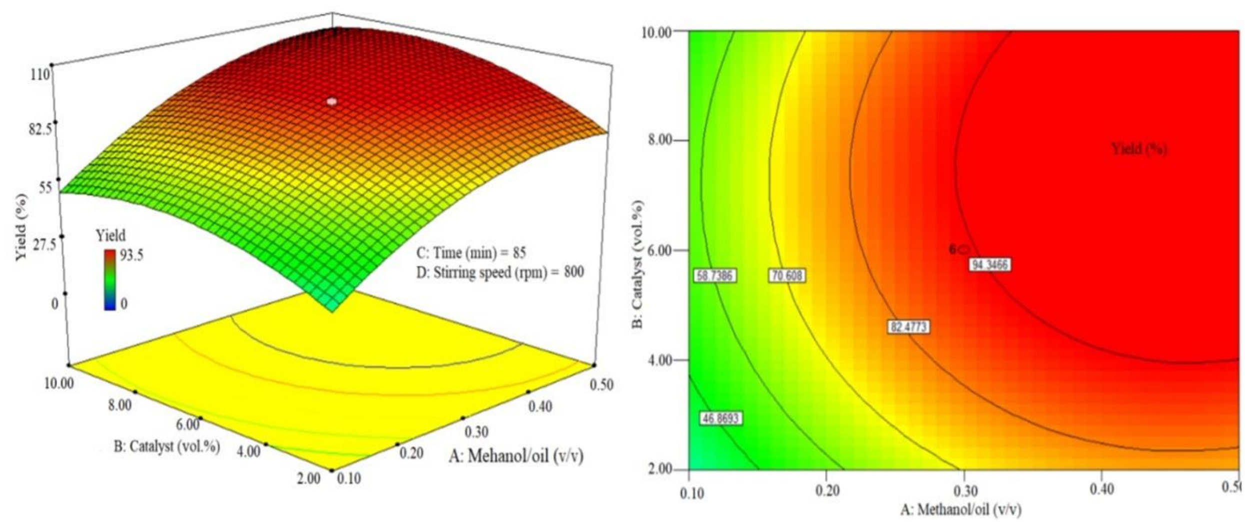

Fig. 6. Influence of methanol and catalyst in the transesterification process for *Ceiba penandra* oil

The influence of the amount of methanol and catalyst concentration during the biodiesel conversion process is depicted in Figure 6 as a 3D surface plot and the accompanying contour plot. While adding more methanol, it appears that the biodiesel yield is gradually rising; nevertheless, as the yield rises more, it becomes virtually saturated. It should be noted that using too much methanol during the conversion process raises the amount of biodiesel produced. Alcohol consumption in excess can increase the solubility in glycerol, which results in poor conversion. The catalyst dosage is increased for Ceiba penandra from 2 wt% to 10 wt% in order to determine the effect of catalyst concentration on biodiesel conversion. However, the yield

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dramatically decreases above a certain level of catalyst addition, and the emulsion that occurs from employing too much catalyst may make biodiesel more viscous.

With the use of a condenser, the temperature is kept constant at 70 °C for efficient conversion. According to Table 1, processing times and stirring rates differ. It is obvious that when processing time is extended, the production of biodiesel increases until it reaches a saturation point. The yield increases as the stirring speed is raised, however if the stirring process is continued, the yield will decrease due to improper mixing. However, in order to maximise biodiesel yield while consuming a minimum amount of energy, the optimum conversion process parameters are obtained statistically from the experimental model [16]. Table 5 lists the optimum process parameter along with the corresponding biodiesel production.

Table 5. The optimized process parameters of transesterification process for maximized biodiesel yield Ceiba penandra

Name of the oil	Methanol/oil (v/v)	Catalyst concentration (wt. %)	Stirring speed (rpm)	Processing time (min)	Biodiesel yield (%)
Ceiba penandra oil	0.34	6	700	85	95.3

The fatty acid profile and other significant characteristics of the biodiesel are used to assess both its quality and quantity [17]. Because saturated fatty acids preserve superior oxidation stability and unsaturated fatty acids and cetane number increase the cold flow qualities, the percentage of saturated and unsaturated fatty acids determines the quality of biodiesel [18]. Figure 7 depicts the fatty acid breakdown of Ceiba penandra biodiesel, which includes 58.1 % saturated fatty acids, 28.2 % monounsaturated fatty acids, and 11.3 % polyunsaturated fatty acids.

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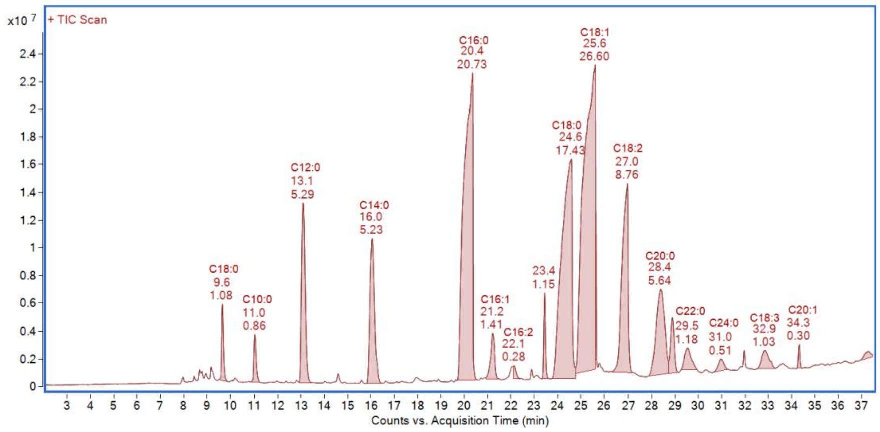

Fig. 7. GC-MS analysis of the biodiesel produced from Ceiba penandra oil

Saturated fatty acids have straight chains of carbon atoms with just one bond, like C14:0, C16:0, and C18:0. On the other hand, the hydrocarbon tail of the carbon chains in unsaturated fatty acids contains one or more double bonds. While polyunsaturated fatty acids have two or more carbon to carbon double bonds, monounsaturated fatty acids (C16:1, C18:1, etc.) only have one bond attached to the CH group (C18:2, C18:3, etc.). The inclusion of 28.2% of a reasonable amount of monounsaturated fatty acids is advantageous because it enhances the qualities of combustion, cold flow stability, and oxidation stability. Additionally, it was claimed to have a significant amount of saturated fatty acids, which can raise the cloud point. A high cetane number is produced by the substantial amount of saturated and monounsaturated fatty acids present in biodiesel, which reduces the ignition delay time and enhances combustion quality [7, 13].

The criteria are summarized in Table 6 and contrasted with the ASTM 6751 and EN 14214 requirements to verify the biodiesel's compliance. The biodiesel's viscosity, cetane number, and cold flow characteristics all affect the length and degree of saturated and unsaturated fatty acids. The majority of the resulting biodiesel's qualities meet the requirements. Kinematic viscosity and density play key roles in fuel injection and engine performance, respectively. A larger droplet can be customised by a higher viscosity, which results in incorrect fuel atomization [19, 20].

Table 6. Ceiba penandra biodiesel properties and comparison with ASTM and EN standards

Properties	Unit	ASTM	EN	*Ceiba penandra* biodiesel
Density at 15ºC	(kg m^{-3})	875 -900	860-900	879
Viscosity at 40ºC	(mm^2 s^{-1})	1.9 - 6	3.5-5.0	4.4

Flash point	(ºC)	Min. 93	Min. 120	142
Total sulphur	(mg kg^{-1})	Max. 15	Max. 10	0.03
Cetane number	-	Min. 47	Min. 51	56
Pour point	(ºC)	-15 to 10	-	8
Oxidation stability,110ºC	(h)	Min. 3	Min.8	5.33
Acid value	(mg KOH g^{-1})	Max. 0.8	Max. 0.5	0.88
Iodine value	-	-	Max. 120	81.2
Phosphorus Content	(mg kg^{-1})	Max. 10	Max. 4	2
Ash content	(% mass)	Max0.02	Max. 0.02	0.00
Cloud point	(ºC)	-3 to 12	-	12
Carbon residue	(% mass)	Max.0.05	Max. 0.3	0.08

Conclusion

Non-edible feedstocks, particularly *Ceiba penandra*, which is widely distributed in South India, are a promising source for the generation of biodiesel. The composition of triglycerides and the FFA content of the initial oil characteristics were determined through analysis. A unique seashell called a Turbo marmoratas was calcined at a temperature of 1000°C, and the calcined powder was then doped with TiO_2, which served as a nanocatalyst for the transesterification process, which is essential for the efficient conversion of biodiesel. Before transesterification, the catalyst was subjected to characterizing by FTIR, XRD, and SEM with mapping in order to demonstrate its superiority. Almost, the oil produced more than 95% biodiesel in the shortest length of time with the least amount of methanol and nano catalyst. To validate its compatibility, the biodiesel's physiochemical parameters are individually checked with ASTM and EN standards. The perfect blend of saturated and unsaturated fatty acids with improved oxidation stability is confirmed by GC-MS analysis, ensuring future applications in the automotive sectors.

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