Acid-based Catalyzed Transesterification Bioprocess for Biodiesel from Jatropha Oil-Based Free Fatty Acids: Its Physical, and Mechanical Properties

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ABSTRACT: *The study investigated the efficiency of biodiesel conversion from jatropha oil-based free fatty acid using an acid-based catalyzed transesterification bioprocess. The maximum Jatropha biodiesel yield was 80% in a 1-hour reaction using a 0.03:1 acid catalyst to oil and a 2:1 alcohol-to-oil ratio. At the same time, the optimum biodiesel yield was in a 2-hour reaction for step 2 by using a 0.03:1 ratio of base catalyst to oil and a 5:1 ratio of alcohol to oil. The Fourier Transform Infrared Spectrometer analysis of Jatropha Biodiesel (JB) revealed a methyl peak (O-CH_3) at 1436.07 cm^{-1}, indicating compatibility with pure mineral diesel, high speed (1295.67rpm), brake horsepower(30.6906KW), mechanical efficiency (53.10%), and lower specific fuel consumption (15.5879 mL/KW. compared with other Jatropha biodiesel blends.*

KEYWORDS: *Free Fatty Acid (FFAs), Biodiesel, Transesterification, Fourier Transform InfraRed (FT-IR) spectrometer, Diesel engine.*

INTRODUCTION

Jatropha curcas Linnaeus (JCL), a small plant native to South America and Africa, was introduced by Portuguese colonists, belongs to the Euphorbiaceae family, and is known as "Physic Nut" [1,2].

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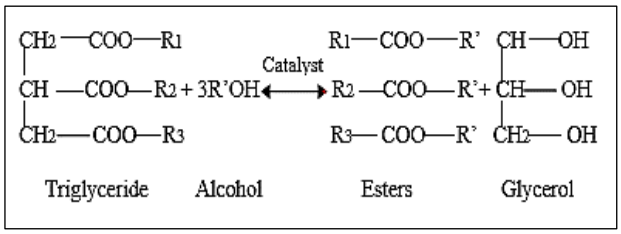

Fig. 1: Transesterification of Triglycerides.

It can grow up to 6 meters tall and can withstand severe droughts [3]. Jatropha seeds contain 30-40% oil and can be propagated through seeds or cuttings [2]. The plant is easy to plant due to its fruit-containing toxin and roots can reduce soil erosion. Fossil fuels are crucial for daily life, producing soap, cosmetics, electricity, and transportation, but their use has harmful impacts on humans, animals, and the ecosystem. Biodiesel, a chemically modified fuel derived from vegetable and animal oils, is a sustainable alternative to non-renewable fossil fuels. Burning fossil fuels releases more than 6 billion tons of carbon dioxide per year, which is twice as much as the biosphere could absorb [4]. Biodiesel is fuel (ester) produced from renewable sources [5-7]. Biodiesel or mono-alkyl esters of long-chain fatty acids is a chemically modified fuel derived from various types of vegetable oils and animal fats [8-12]. The feedstocks for biodiesel production are seed oils like sunflower, soybean, castor, rapeseed, and palm oil [13-17]. The study aimed to determine the optimal alcohol-to-oil and catalyst-to-oil ratios for producing high-quality Jatropha biodiesel (JB). The production methods, equipment, material, temperature, and reaction time were verified through laboratories. The Jatropha oil-free fatty acid value was measured before and after the esterification process. The biodiesel was analyzed using a Fourier Transform Infrared Spectrometer and tested in a diesel engine. The current commercial production technology of biodiesel through homogeneous transesterification has a lot of limitations which make the cost of biodiesel production economically unfeasible. Alcohol combines with the triglycerides of fatty acids (vegetable oil) in the presence of a catalyst during the transesterification process, a valuable and well-established chemical reaction equation that produces glycerol and esters [19–21]. Using a Fourier Transform Infrared Spectrometer (FT-IR), the produced Jatropha Biodiesel (JB) was examined and its composition was contrasted with that of mineral diesel. The biodiesel produced was tested as a blend in a diesel engine. The diesel engine performance was studied by calculating the diesel engine performance parameters using different blends.

Figure 1 shows the transesterification of triglycerides. This research aimed to investigate the effectiveness of employing acid-based transesterification to produce biodiesel from crude Jatropha oil and assess diesel engine performance and physical and chemical properties.

EXPERIMENTAL SECTION

Oil extraction technique

Soxhlet Extractor- The process involved grinding Jatropha seeds, filling them into a thimble, filling n-hexane (3/4 of the flask) with boiling chips or beads, and letting it cool. After eight hours, the solvent was removed and the amount of Jatropha oil recovered was calculated. The percentage of Jatropha content was then calculated.
Mass of Jatropha oil = (weight of the flask + boiling chips + extracted oil) – (weight of the flask + boiling chips). Jatropha content (%) = [mass of Jatropha oil extracted (g)/ sample weight (g)] x 100%.

The oil Expeller (Piteba) was a method for extracting pure Jatropha oil from seeds. It involved drying the seeds, sifting them, heating the press cage, filling the funnel, and turning the crank. The oil was collected, cleaned, and left at room temperature for a few hours. The oil was filtered to obtain pure Jatropha oil.

Free fatty acids measurement

Titrimetry was used to determine the acid value of vegetable oils, which was the number of mg of KOH needed to neutralize fatty acids in 1 gm of fat. Reagents included a solvent mixture, 0.1 M KOH in ethanol, and phenolphthalein in 95% ethanol. The oil was weighed, a solvent mixture was added, and heated on a hot plate. The oil was titrated with KOH solution, and the acid value was calculated using a formula (56.1 x M x V)/ W. If the acid value exceeded 4%, sulphuric acid was used as a catalyst in the pre-treatment process, and if it was less than 4%, biodiesel production proceeded.

Materials and equipment used

The research was carried out in the Faculty of Engineering at the University Malaysia Sarawak and used various materials to produce biodiesel, including methanol, MeOH, KOH, sulphuric acid, H_2SO_4, crude

Table 1: Shows titration results.

Trail	First	Second	Third
W/g	7.45	8.62	5.32
V/ml	38.5	44	28.50
Acid Value	28.98	28.63	30.0

Fig. 2: Esterification using reflux and water bath method.

Jatropha oil (CJO), and various equipment. The two-step acid-based transesterification method was found most suitable.

One-step-based catalyzed transesterification

The process involves filtering CJO, heating it to over 100ºC, and preparing methoxide by mixing methanol with potassium hydroxide. The methanol to oil ratio (1:1, 2:1, 3:1, 4:1, 5:1 & 6:1) and potassium hydroxide amount was determined by adding the number of KOH ml used during titration and 5.0. The mixture was heated (50ºC) and stirred using a magnetic stirrer (350 rpm) for 2 hours, then cooled and agitated using an orbital shaker for 2 hours at room temperature. The mixture was then filtered, separated into layers, and washed with desterilized water. The filtered biodiesel was then heated to remove water content and produced pure biodiesel.

Two-Step Acid-Based Catalyzed Transesterification

First step: acid-catalyzed esterification

The process of esterification involved filtering and heating CJO to remove high FFA, followed by heating a mixture of sulphuric acid and methanol at 50ºC. The study examined various sulphuric acid to oil ratios to determine the optimal ratio (0.01:1, 0.02:1, 0.03:1, 0.04:1, 0.05:1 and 0.06:1), with the reflux and water bath methods shown in Figure 2. The optimum methanol-to-oil ratio was found, and the mixture was then poured into the heated CJO,

Fig. 3: The endpoint of the titration.

allowed to react for one hour, and then washed to remove impurities and sulphuric acid. The oily product was heated to 100°C for water removal.

Second step: base-catalyzed transesterification

In the second step, the oily product was heated from step 1 at 50ºC until it turned into clearer fluid. The mixture of potassium hydroxide and methanol was heated until it reached 50ºC before being added to the heated oily product and stirred the reaction mixture at 65ºC for 2 hours. After that, the complete reaction mixture was allowed to settle overnight, and then removed the lower layer of glycerol.

RESULTS AND DISCUSSION

Table 1 shows the acid value and Fig. 3 shows the end point of titration.

One-step-based catalyzed transesterification

The transesterification method for Jatropha biodiesel produced low yield due to high free fatty acid content in crude Jatropha oil, limited conversion, and soap formation. The two-step transesterification method was applied to determine the optimal ratio of methanol to oil and catalyst to oil while studying the impact of equipment and reaction time.

Two-Step Acid-Based Catalyzed Transesterification

Since crude Jatropha oil-free fatty acid was more than 4%, the two-step transesterification method was applied. During this two-step method, the methanol-to-oil and catalyst-to-oil optimum ratio needed to be determined. The influence of equipment and time of reaction variables on the biodiesel production yield was studied through the laboratory.

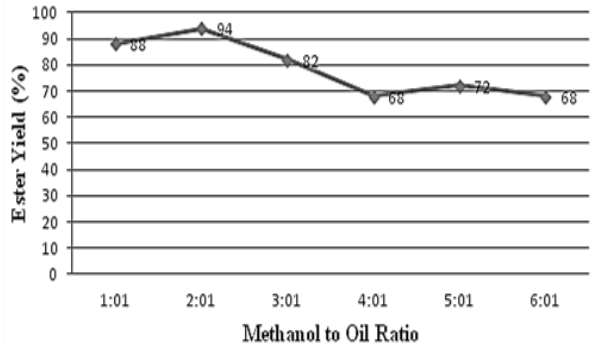

Fig. 4: Ester yield by changing methanol to oil ratio for step 1.

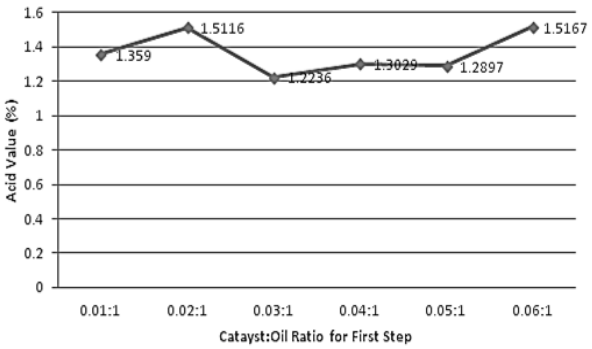

Fig .6: Acid value for step 1 with different catalyst to Oil ratios.

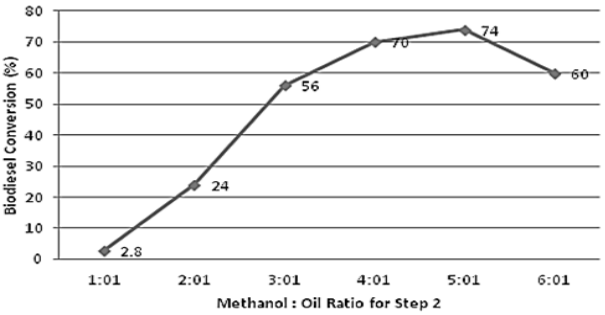

Fig. 5: Biodiesel production by changing methanol to oil ratio for step 2.

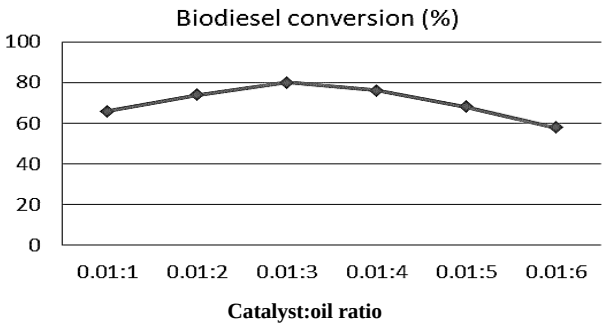

Fig. 7: Biodiesel production by changing catalyst to oil ratio for step2.

Methanol to oil ratio

The study tested methanol-to-oil ratios from 1 to 6 and found the optimal ratio at 2:1 for the highest ester yield (94%) (Fig.4).

Utilization of excess methanol reduced yield made it wasteful, and was not economically practical. Therefore, determining the optimal methanol-to-oil ratio was crucial for the highest yield. It was reported that a similar biodiesel yield was observed with the same ratios [23-24]. The optimum methanol to oil ratio for the second step in biodiesel production was 5:01. Excess methanol after this ratio reduced biodiesel yield, making it economically unpractical to use excess methanol without finding the optimum ratio (Fig. 5). This highlighted the importance of methanol in the process.

Catalyst to oil ratio

The esterification process used sulphuric acid as a catalyst to convert free fatty acid into ester (Fig. 6).

The optimum catalyst-to-oil ratio was determined to have the lowest acid value, aiming to remove crude Jatropha oil-free fatty acid below 4%. The titration method determined the optimal catalyst for oil, resulting in an optimum ratio of 0.03:1 or 3%

sulphuric acid, ensuring all esters were ready for biodiesel production. The biodiesel conversion rate increased until a base catalyst-to-oil ratio of 3:01, with the maximum Jatropha biodiesel yield of 80% (Fig. 7).

The addition of a base catalyst did not improve efficiency but reduced yield. The efficiency of magnetic stirrers ranges from 68-72%, while orbital shakers have an average efficiency of 78.67%. The mixing rate was more important than temperature in biodiesel production, and the orbital shaker was chosen for laboratory use. The highest ester conversion yield is 92% at a one-hour reaction.

Type of equipment

According to Figure 8, the biodiesel conversion efficiency of the magnetic stirrer (350 rpm) was between 68–72%, with an average efficiency of 69.33%.

However, the biodiesel conversion efficiency using an orbital shaker had fallen between 76–80% with an average value of 78.67%. Therefore, compared to a magnetic stirrer/cum heater, the biodiesel conversion efficiency achieved using an orbital shaker was greater. Compared to the magnetic stirrer/cum heater, the orbital shaker had a higher and more balanced mixing rate.

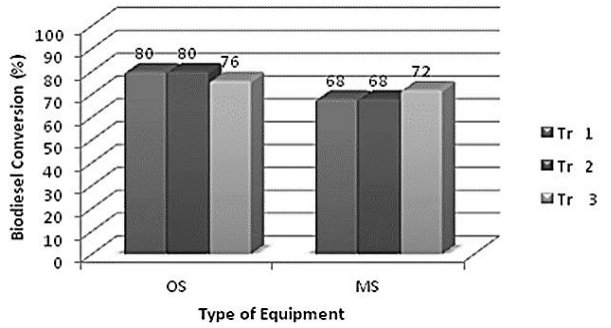

Fig. 8: Biodiesel conversion by changing the type of equipment used (Tr: Trial).

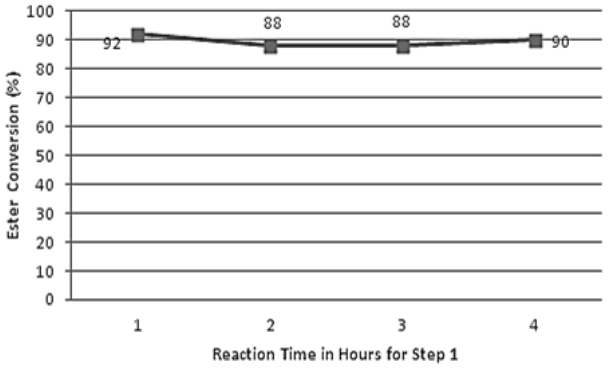

Fig. 9: Ester yield by changing reaction time for step 1.

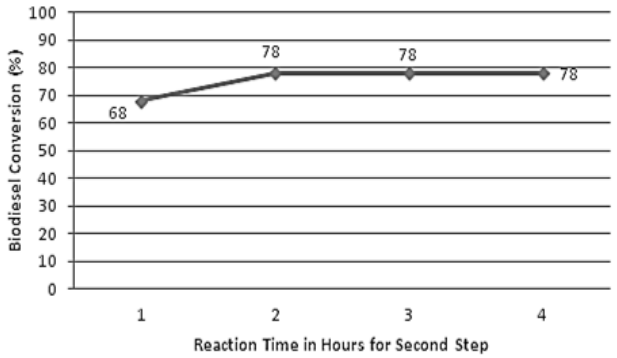

Fig. 10: Biodiesel yield by changing reaction time for step 2.

As a result, biodiesel yield relied on the combination rate; an unbalanced mixing rate reduced yield due to an insufficient conversion process. An orbital shaker can manufacture biodiesel without heating at room temperature.

This finding demonstrated that the mixing rate was more significant than the temperature in producing biodiesel. Thus, the orbital shaker was selected as the biodiesel manufacturing apparatus in the laboratory. Figure 9 shows that the ester conversion yield was the greatest at 92% in an hour-long process.

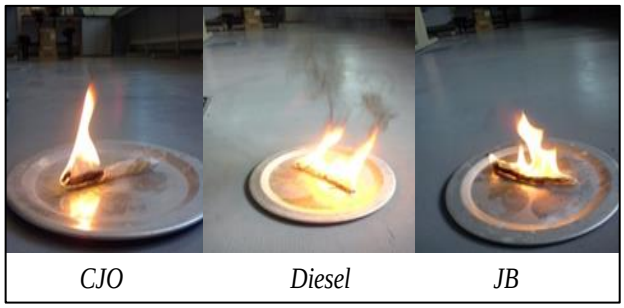

Fig. 11: Smoke observation by direct burning CJO, diesel, and JB.

The ester yield dropped from 92% to 88% with increased reaction time. Since the esterification process was reversible, it would revert from ester to crude oil if the reaction was not kept at a consistent temperature or took too long.

$$\text{CJO + MeOH} \overset{catalyst+\Delta}{\longleftrightarrow} \text{Ester + water}$$

The study showed that the biodiesel yield remained consistent at 78% even with a two-hour reaction time, despite an increase to four hours (Fig. 10). The biodiesel and crude Jatropha Oil (CJO) exhibited light smoke emission, while diesel produced black dark smoke. The FT-IR spectrum of JB and CJO showed a methyl peak ($1436.0cm^{-1}$) in JB, while CJO did not show.

The diesel spectrum did not show this C=O bond, and the ester carbon-oxygen ($1260\text{-}1000cm^{-1}$) and carbon-hydrogen (2960 to $2850cm^{-1}$) bond peak frequencies were different [25-26].

Burning test on Jatropha Biodiesel (JB), Crude Jatropha Oil (CJO), and mineral diesel

The oil emission was tested and monitored using the burning test. As it can be seen with our own eyes, Fig. 11 shows that while the CJO and JB emitted light smoke, the diesel emitted a large amount of dense, black smoke. The JB burned entirely without emitting any dark smoke, unlike diesel.

Jatropha Biodiesel (JB), Crude Jatropha Oil (CJO), and diesel spectrum analysis

FT−R graph of JB and CJO is shown in Figure 12. The spectrum range was $70cm^{-1}$ to $4000cm^{-1}$. The main difference between these two spectrums, as methyl peak ($O\text{-}CH_3$), exhibited in the JB spectrum at $1436.0cm^{-1}$, but CJO spectrum did not show this peak. The methyl esters of every kind of fatty acid present in the biodiesel were displayed using this peak.

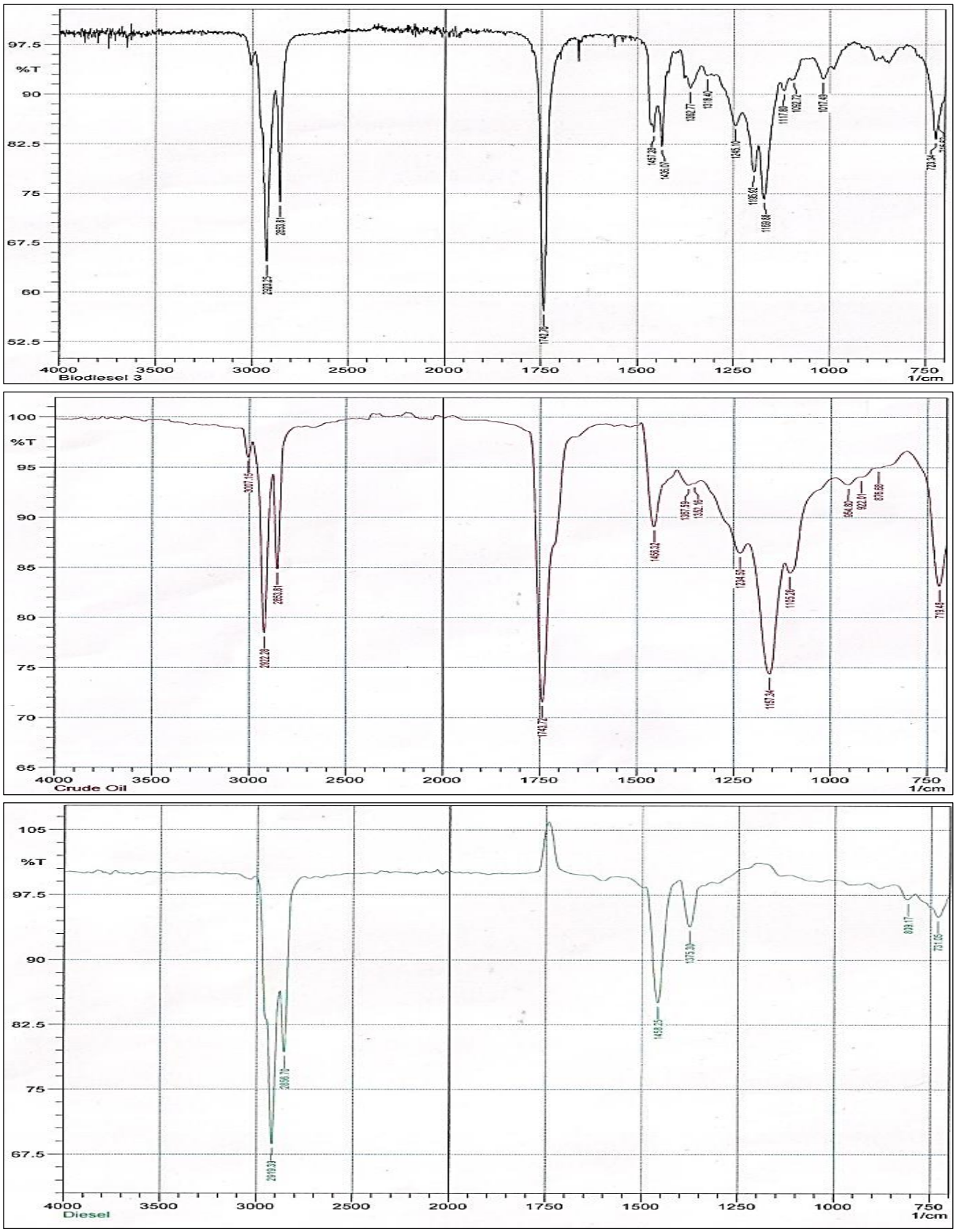

Fig.12: FT-IR Spectrum for (i) JB, (ii) Crude Jatropha Oil, (iii) Diesel.

Table 2: Diesel engine performance parameters.

Parameters	Formula
Torque (T)	T = Fxr
Fuel Consumption Rate (FCR)	FCR = v/t
Engine Power Output (P)	(IV Cos π) / 1000
Specific Fuel Consumption (SFC)	V/p
Brake Horsepower (Bhp)	$2\pi vT$
Indicated Horsepower (Ihp)	NplAn / 1000
Mechanical Efficiency (η)	(Bhp/Ihp) ×100%

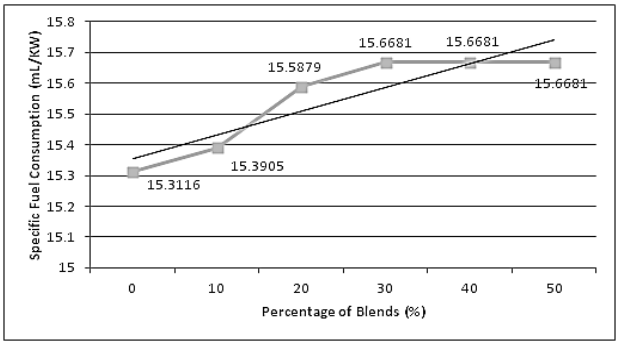

Fig. 13: Specific Fuel Consumption (SFC) versus percentage of blends.

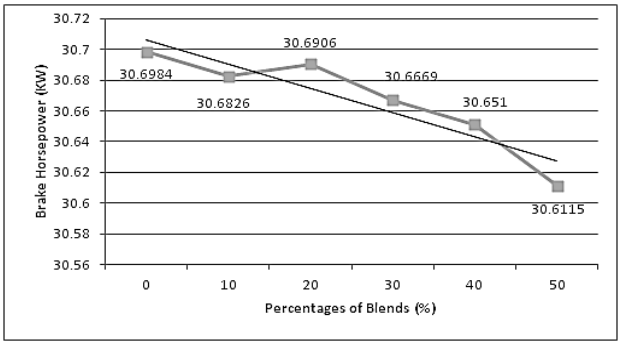

Fig .14: Brake horsepower of diesel engine testing using different JB blends.

Because of its ester carbonyl bond (C=O) or ester for both spectrums, FAME exhibited the highest infrared absorption at 1741–1745 cm^{-1}. Nonetheless, this C=O bond was absent from the diesel spectrum.

Diesel engine performance study

The study tested Jatropha biodiesel blends in a diesel engine, starting with a warm-up period using mineral diesel (Table 2). Specific fuel consumption increased with the Jatropha biodiesel blend percentage, with mineral diesel having the lowest specific fuel consumption. Brake horsepower increased with Jatropha biodiesel blend percentage, with the optimum point at 30.6906kW. However, mechanical efficiency decreased with the Jatropha biodiesel blend percentage, with B_{20} being the optimum blend.

Engine speed and mechanical efficiency decreased with the Jatropha biodiesel blend percentage but increased with engine speed. The oily product yield was 1480mL or 98.67% after the first step esterification process, and 700mL or 70% for the second step transesterification process. From Figure 13-15, specific fuel consumption increased from B_0 to B_{30} and then it was constant until B50. The mineral diesel had the lowest specific fuel consumption because the diesel engine used less fuel to generate 1kW of power than other blends.

The SFC value increased with increasing Jatropha biodiesel blend percentage.

The brake horsepower value was basically decreasing but it increased at B20. So, the optimum brake horsepower point was located at B_{20} with a value of 30.69kW. Figure 14 showed the engine power decreased with increasing the JB blend. Mechanical efficiency was measured which represented the effectiveness of an engine in transforming fuel energy to mechanical work in percentage.

Mineral diesel showed the highest mechanical efficiency (53.11%) and B_{50}showed the lowest mechanical efficiency (52.96%). The line graph was generally decreasing trend but it increased at $B_{20.}$ So, $B_{20}$was the optimum blend. The diesel engine speed (rpm) versus mechanical efficiency (%) for different JB blends are shown in Figure 16.

Engine speed and mechanical efficiency decreased with increased JB blend percentage and mechanical efficiency increased simultaneously with engine speed. The highest speed occurred at B_0 with a value of 1296rpm and the lowest speed was located at B_{50} with a speed of 1292.33rpm.

However, at the B_{20} blend, the speed and the mechanical efficiency increased to 1295.67rpm and 53.1%. The result showed that B_0 and B_{20} had almost similar diesel engine performance.

Hence, B_{20} was the optimum blend which had the power output almost similar to B_0.

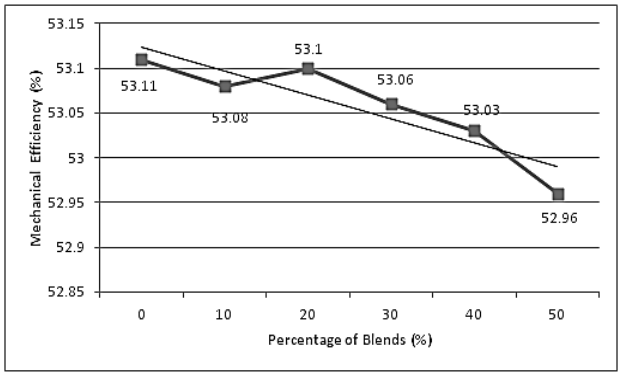

Fig. 15: Mechanical efficiency of diesel engine testing for different JB blends.

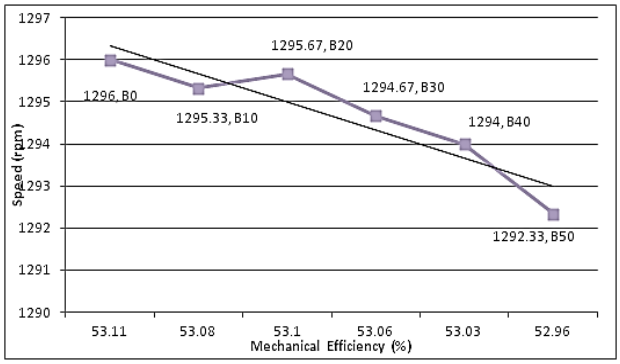

Fig. 16: Diesel engine speed (rpm) versus mechanical efficiency (%) for different JB blends.

CONCLUSIONS

The two-step transesterification method was found to be more suitable for removing free fatty acid (FFA) in crude Jatropha oil, with the water bath and reflux method being more suitable. The optimum oil-to-methanol ratio was 1:2, and the optimum oil-to-catalyst ratio was 1:0.03. An orbital shaker produced a higher oil yield than a magnetic stirrer. The maximum yield of Jatropha biodiesel was 80%. The B_{20} blend had similar brake horsepower and mechanical efficiency as mineral diesel, providing that diesel engines run smoothly with the Jatropha biodiesel blend.

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