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# Optimization and Characterization of Acid-Catalyzed Castor Biodiesel and its Blends

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**Abstract:** In terms of energy security, biodiesel has become an alternative, safe, and biodegradable fuel. Here, to produce biodiesel from castor oil, a transesterification process was carried out using an acid-catalyzed catalyst. Three blends ($B_{10}$, $B_{20}$ and $B_{30}$) were prepared by using different proportions of castor biodiesel and petro-diesel. Biodiesel optimum yield of 80% was obtained from 5 mL of castor oil with the influence of different parameters such as 1.75 mL of methanol and 0.08 mL of conc. $H_2SO_4$, at 65 ℃, for 3 hours with 600 revolutions per minute stirring speed for 5 mL of castor oil. Physiochemical properties of all samples such as moisture contents, iodine value, free fatty acid value, saponification value, ester value, acid value, peroxide value, viscosity (at 40 ℃), specific gravity, refractive index, density, boiling point, average molecular weight, and higher heating value were determined. Castor biodiesel characterization was resulted as 0.112 (percentage), 86 mg KOH/g, 1.0878 mg KOH/g, 0.439 mg KOH/g, 84.9122 mg KOH/g, 86.32 g$I_2$/100 g, 20.66 Meq/100 g, 0.8850 g, 17.21 cSt, 1.4667 nD, 0.8910 g, 290 ℃, 1982.05 g, and 44.479 MJ/Kg min, respectively. The functional groups were investigated by using FTIR. In the present study, it was demonstrated that biodiesel can be produced using a method of acid-catalyzed transesterification by using castor oil.

**Keywords:** Biodiesel, castor oil, transesterification, optimization, iodine value, saponification.

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## INTRODUCTION

As the population grows and energy consumption increases in the industry, agriculture, domestic and public sectors, the energy crisis has become a major problem for the world. (1). At present, energy security has become a central issue because energy demands per capita are also increasing day by day (2). Government of Pakistan taking multiple steps to overcome the energy crisis and has been creating friendly relations with fuel-rich Muslim countries such as Saudi Arabia, UAE, Qatar, Iran etc. Excessive investment has been done in the country's oil refineries. Searching for new and renewable energy such as biodiesel is one of the alternative ideas to put back fossil fuels in the present situation (3). The blending of fossil fuels with biodiesel controls exhaust emission, moreover it is a green, cheaper, eco-friendly, and easily producible energy source (4). Pakistan is an energy-deficient country and facing an energy crisis because of limited fossil fuels (5). On the other hand, public demands for energy consumption are increasing due to rapid transportation, industrialization, agriculture, and household usage. At present, China has the highest demand in the continent of Asia (6). As world oil prices are continuously rising, developing countries like Pakistan need a cheaper source of energy (7). There is a continuous increase in the cost of energy in Pakistan, which is expected to peak after 2050. A minimum share of 5% of total diesel consumption must be achieved by blending biodiesel with petroleum diesel under current energy sector policies (8). Today, like Pakistan

whole world is facing energy crisis, petrol, electricity, and gas prices increasing more rapidly. In Spain, electricity price raised up to 200% in 2022 while in India price increased 110% more than the last year.

Biodiesel is known as oxygenated fuel having similar properties to diesel and normally can be produced from waste cooking oil, animal fats, and vegetable oils by conversion of triglycerides to esters via transesterification (9). It is a non-toxic, clean, bio-degradable, cheaper, technologically feasible, and renewable fuel (10). It can also use directly in the diesel engine but using directly is not suitable for the efficiency of the engine due to high viscosity, poor combustion, and non-volatility (11). Therefore, some modifications are necessary to oil like high viscosity of oil can be reduced by preheating, transesterification, blending, and thermal cracking. Most commonly, reducing the viscosity of oil transesterification is preferred (12). According to literature, 10% of biodiesel is transesterified (13). The process is done by the reaction of triglycerides with short-chain alcohol usually methanol or ethanol by using a catalyst resulting alkyl esters (biodiesel) and glycerin (14,15).

As oil is a triglyceride of fatty acid and glycerol and the most widely used feedstock is vegetable oils for the production of biodiesel. Due to the shortfall of edible oil it would not be executable to develop biodiesel from edible oil. Moreover, edible oil is also used for cooking purposes (16). So it is necessary to produce biodiesel from non-edible oil resources such as castor oil, atrophy oil, neem oil, Karana etc., (17-19). Castor biodiesel is a non-edible, versatile and renewable energy source that replaces the petroleum-derived diesel fuel and can act as lubricant (20,21). Castor-oil is extracted from castor beans and contain 40-55% oil while other commonly used crop seeds contain a low concentration of oil like soybean 15–20%, palm 30-50%, sunflower 25–35% and rapeseed 38–46% (22). Castor oil contains hydroxylated fatty acid 80-90% mainly ricinoleic acid and non-hydroxylated fatty acids approximately 10% mainly 4-5% linoleic acid, 2-4% oleic acid, 1% stearic acid and 1% palmitic acid (23). In comparison with other vegetable oils, castor oil has a higher cetane number since it does not contain sulfur, indicating it contains more oxygen and is more flammable (24). Castor oil, due to the presence of ricinoleic acid, is approximately 7 times more viscous than other vegetable oils which increases the lubricity of biodiesel (25). Due to its low pour point -45℃ and cloud point, castor biodiesel is the best fuel for cold weather because it has a low pour point and cloud point (26).

For transesterification of castor oil several methods have been reported by using homogeneous catalysts and heterogeneous catalysts. Panwar *et al* by using an alkaline catalyst produced 96% biodiesel from castor oil via transesterification (27). Jeong & Park and Thirugnanasambandham *et al* synthesize biodiesel using KOH as a catalyst with a yield up to 92% and 86.9% (28,29). Nurdin *et al* studied the transesterification by using a heterogeneous catalyst (calcined mussel shell) yielding 91.17% (30). Amalia *et al* used heterogeneous KOH/zeolite catalyst for the production of castor biodiesel through the transesterification process (31). Ferdous *et al* used acid catalyst (sulfuric acid) for the production of biodiesel with a yield of more than 70% (32).

In this study, transesterification of castor oil was performed by using acidic catalyst sulfuric acid with an optimum yield of 80%. The physiochemical properties like saponification value, moisture contents, acid value, ester value, iodine value, peroxide value, free fatty acid value, specific gravity, viscosity (at 40 ℃), refractive index, density, boiling point, average molecular weight and higher heating value were studied. Furthermore, the functional groups were investigated by FTIR.

## MATERIALS AND METHODS

### Collection of feedstock

Castor seeds were purchased from the local market of Bahawalpur, Pakistan, washed to remove dirt, dried for 4 days in sunlight and finally in the oven for 5 hours at 100℃ to remove water. Castor oil was extracted by a conventional extractor and filtered through Whatman filter paper to remove suspended particles.

### Transesterification experiments

As free fatty acid (FFA) value of castor oil is very high and can cause a major problem in the preparation of biodiesel. To overcome this problem, acid-catalyzed transesterification can be done.

40 mL of castor oil was heated for 5 min with vigorously stirring. A mixture of 14 mL of methanol and 0.64 mL of conc. $H_2SO_4$ was added to hot castor oil. The temperature was adjusted to 65 ℃, the mixture was properly covered to control the loss of methanol and stirred with a magnetic stirrer continuously for 3 hours. After the completion of the reaction, the mixture was allowed to settle for 24 hours in 250 mL of separator funnel. After 24 hours two layers were formed, the upper layer was methyl ester (biodiesel) and lower the layer was glycerol & gums. The lower layer was separated safely, without the loss of the upper layer. The biodiesel layer was washed with hot water several times. After washing the biodiesel layer in the separator funnel, 15-20 mL of boiled distilled water was added into the separator funnel and allowed to stand for another 6 hours which resulted in the remaining traces of glycerin setting down into the

water. After 6 hours, water was removed and separated the biodiesel layer into a beaker. Biodiesel was heated above 100℃ to remove the moisture contents. Finally, the biodiesel was prepared and stored for further usage. The biodiesel percentage yield was measured by using the formula:

$$Biodiesel\ \%age\ yield = \frac{volume\ of\ biodiesel\ obtained\ (mL)}{volume\ of\ raw\ castor\ oil\ (mL)}\ x\ 100 \qquad \text{(Eq. 1)}$$

## RESULTS AND DISCUSSION

In transesterification reaction, triglyceride which is a mixture of fatty acids react with alcohol in the presence of a catalyst (acid or base). In this reaction alkyl ester (biodiesel) is produced and glycerol is obtained as a by-product (Figure 1).

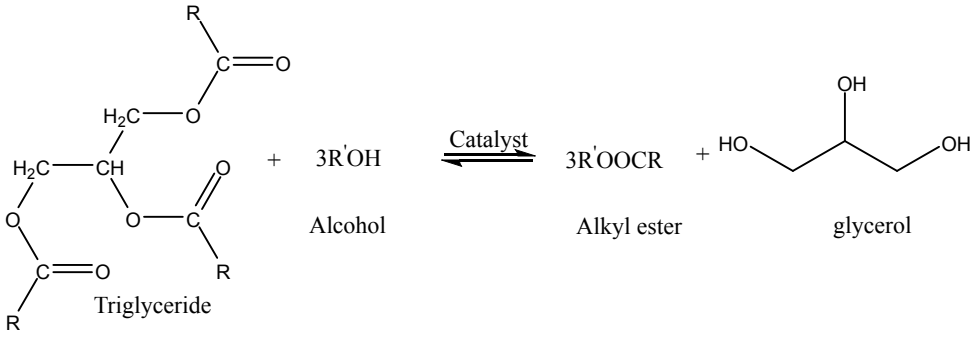


**Figure 1:** Transesterification reaction of triglyceride.

For high free fatty acids and moisture content feed-stocks, the use of acid catalyst is favorable as compared to alkali catalysis. By acid catalyst, free fatty acid is reduced and oil can convert into biodiesel. Sulfuric acid, orthophosphoric acid, and hydrochloric acid are among mostly used acid catalysts. The reaction of free fatty acid for the extraction of biodiesel involves the absorption of FFA on catalyst acidic position which forms carbocation. An intermediate is produced by the reaction of methanol with carbocation. By elimination of water from intermediate biodiesel is produced as an end product (Figure 2).

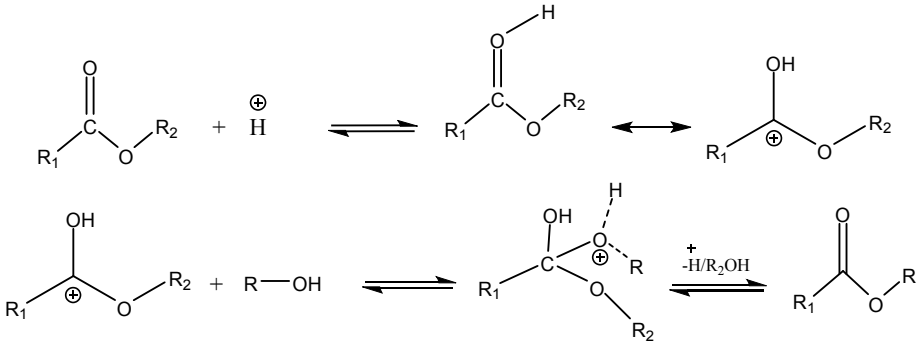

**Figure 2:** Acid-catalyzed transesterification mechanism.

### Effect of variable parameters

The effect of methanol concentration, conc. $H_2SO_4$ catalyst concentration, reaction temperature, reaction time and the stirring speed was investigated. The data is summarized in Table 1.

**Table 1:** Optimization Summary of castor biodiesel

| Castor oil | Methanol | $H_2SO_4$ catalyst | Reaction temperature | Reaction time | Stirring speed | Biodiesel percentage |
|---|---|---|---|---|---|---|
| ***Effect of methanol concentration*** | | | | | | |
| **20 mL** | 5 mL | 0.32 mL | 65℃ | 3 hours | 600 rpm | 67.5% |
| **20 mL** | 7 mL | 0.32 mL | 65℃ | 3 hours | 600 rpm | 77.5% |
| **20 mL** | 10 mL | 0.32 mL | 65℃ | 3 hours | 600 rpm | 64% |
| **20 mL** | 12 mL | 0.32 mL | 65℃ | 3 hours | 600 rpm | 56% |
| ***Effect of catalyst concentration*** | | | | | | |
| **10 mL** | 3.5 mL | 0.16 mL | 65℃ | 3 hours | 600 rpm | 67% |
| **10 mL** | 3.5 mL | 0.25 mL | 65℃ | 3 hours | 600 rpm | 60% |
| **10 mL** | 3.5 mL | 0.32 mL | 65℃ | 3 hours | 600 rpm | 50% |
| **10 mL** | 3.5 mL | 0.50 mL | 65℃ | 3 hours | 600 rpm | 39% |
| ***Effect of reaction temperature*** | | | | | | |
| **5 mL** | 1.75 mL | 0.08 mL | 45℃ | 3 hours | 600 rpm | 48% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 600 rpm | 66% |
| **5 mL** | 1.75 mL | 0.08 mL | 85℃ | 3 hours | 600 rpm | 58% |
| **5 mL** | 1.75 mL | 0.08 mL | 105℃ | 3 hours | 600 rpm | 0% |
| ***Effect of reaction time*** | | | | | | |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 1 hour | 600 rpm | 0% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 2 hours | 600 rpm | 28% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 600 rpm | 42% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 4 hours | 600 rpm | 0% |
| ***Effect of stirring speed*** | | | | | | |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 300 rpm | 0% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 400 rpm | 28% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 500 rpm | 42% |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 600 rpm | 0% |
| ***Optimum conditions for all parameters*** | | | | | | |
| **5 mL** | 1.75 mL | 0.08 mL | 65℃ | 3 hours | 600 rpm | 80% |

**Effect of methanol concentration**

Four transesterification reactions were performed by using 20 mL of castor oil and variable concentration of methanol such as 5 mL, 7 mL, 10 mL & 12 mL, catalyst (0.32 mL of conc. $H_2SO_4$), reaction temperature (65 ℃), reaction time (3 hours) and stirring speed (600 rpm). Obtained biodiesel percentage yield were 67.5%, 77.5%, 64% and 56% respectively. Phase separation was also done for all given concentrations of methanol. Below 5 mL of methanol concentration, the reaction was not proceeding and phase separation was not observed due to the low concentration of methanol not equilibrating with castor oil. Hence transesterification did not proceed.

It was noticed that a maximum of 77.5% of biodiesel yield was obtained by using 7 mL of methanol. For acid-catalyzed transesterification, biodiesel percentage yield was decreased as the methanol concentration was increased. These changes resulted that excess methanol lowered the biodiesel yield because of increasing the miscibility of castor oil into excess methanol at a given reaction conditions. A high concentration of methanol increased the solubility of castor oil into methanol. –OH group present in ricinoleic acid makes the castor oil polar. On the other hand, a solvent such as methanol is also polar due to the presence of the -OH group. So, according to the principle "like dissolves like" methanol shows the miscibility into castor oil. That's why biodiesel yield was decreased. Finally, 7 mL of methanol concentration were considered as the optimal value of methanol for 20 mL of castor oil. Keera *et al* (2018) reported the similar results to our findings, increasing the methanol to oil ratio led to decreased biodiesel yield as a result of methanol accumulation and viscous fluids. Figure 3 represents the biodiesel yield at different methanol concentrations.

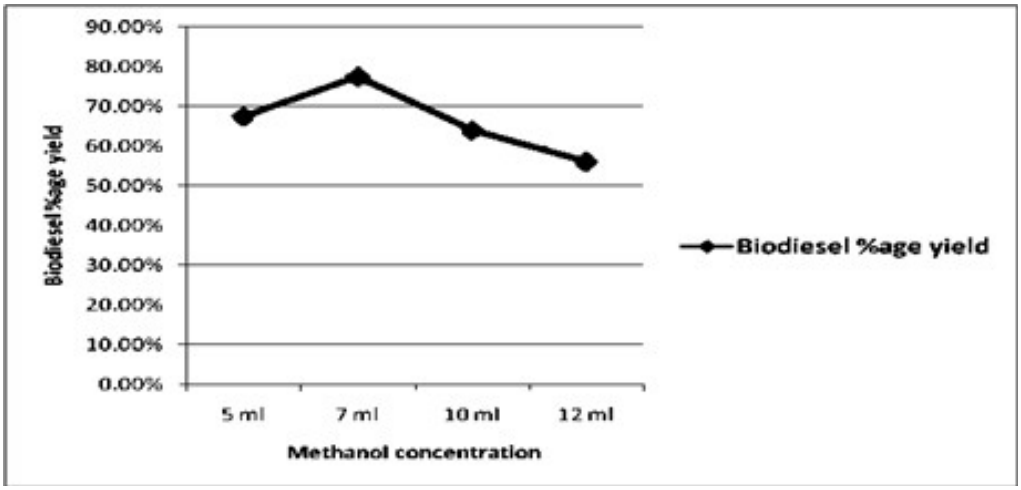


**Figure 3:** Effect of methanol concentration on biodiesel percentage yield.

**Effect of conc. $H_2SO_4$ catalyst concentration**

The acidic catalyst was used for transesterification because the basic catalyst caused emulsion and saponification. Four transesterification reactions were performed by using 10 mL of castor oil and a variable concentration of conc. $H_2SO_4$ catalyst such as 0.16 mL, 0.25 mL, 0.32 mL, and 0.5 mL; methanol concentration, reaction temperature, reaction time, and stirring speed was 7 mL 65 ℃, 3 hours, and 600 rpm, respectively. Obtained biodiesel percentage yields were 67%, 60%, 50%, and 39%. Below 0.16 mL of conc. $H_2SO_4$ catalyst, transesterification reaction did not proceed and phase separation was not achieved.

It was observed that maximum of 67% of biodiesel yield were obtained by using 0.16 mL of conc. $H_2SO_4$ catalyst. Chand *et al* (2013) was also reported a 68% yield by using a 0.5% KOH catalyst (33). But when using KOH catalyst, chances of saponification is higher than the use of conc. $H_2SO_4$ catalyst. That is why we used conc.$H_2SO_4$ catalyst to overcome saponification. Biodiesel percentage yield decreased as the conc. $H_2SO_4$ catalyst increased. These changes resulted in a high volume of acidic catalyst that lowered the biodiesel yield. All this is because of high free fatty acid (FFA) value of castor oil tends to resist biodiesel production. Conc. $H_2SO_4$ catalyst converted the free fatty acid into biodiesel but over volume of conc. $H_2SO_4$ catalyst decreased the biodiesel yield because the catalyst start mixing with glycerol and spoil the biodiesel during transesterification. So, an adequate volume of conc. $H_2SO_4$ catalyst is efficient to produce the biodiesel. Finally, 0.16 mL of conc. $H_2SO_4$ catalyst were considered as the optimal value of acidic catalyst for 10 mL of castor oil. Carmaker *et al* (2018) agrees with our findings (34). Figure 4 represents the biodiesel yield at different conc. $H_2SO_4$ catalyst concentrations.

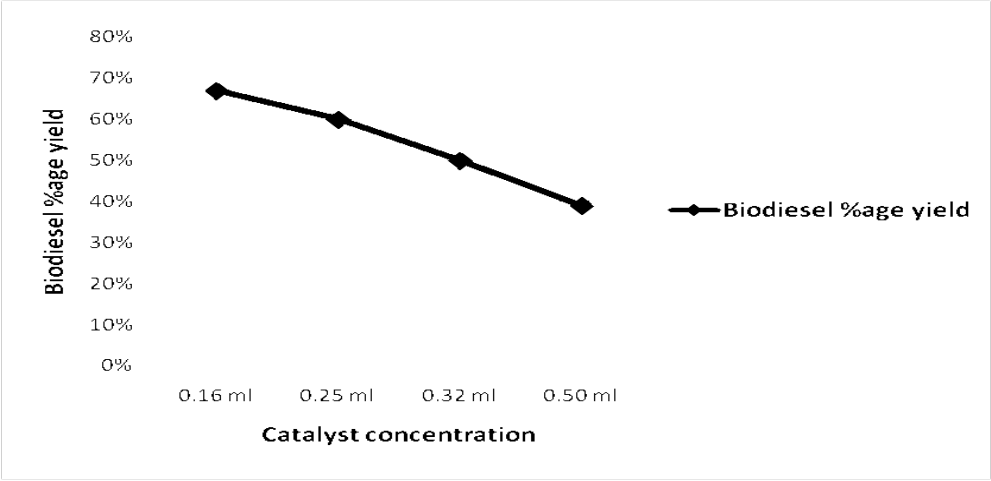


**Figure 4:** Effect of conc. $H_2SO_4$ catalyst concentration on biodiesel percentage yield.

**Effect of reaction temperature**

Four transesterification reactions were performed by using 5 mL of castor oil and variable reaction temperatures such as 45 ℃, 65 ℃, 85 ℃, and 105 ℃. The methanol concentration, conc. $H_2SO_4$ catalyst concentration, reaction time, and stirring speed were 1.75 mL, 0.08 mL, 3 hours & 600 rpm, respectively. The percentage yield of obtained

biodiesel was 48%, 66%, 58% and 0% respectively. Phase separation was performed at 45 ℃, 65 ℃, and 85 ℃ of temperature but not done at 105 ℃.

It was observed that a maximum of 66% of biodiesel yield was obtained at 65 ℃. But minimum biodiesel yield was obtained at 45 ℃. These changes resulted, at 105℃ of high-temperature castor oil contents being dissolved into methanol. That is why no phase separation took place at 105 ℃. At 45 ℃ of temperature phase separation was done but the yield was low because of incomplete transesterification. Finally, 65 ℃ of reaction temperature was considered the optimal value for transesterification. Maryam *et al* (2018) reported that increased in temperature from 60 ℃, biodiesel yield decreased (10). Similar results were reported by Silting *et al* (2016) that above 65 ℃ biodiesel yield decreased due to the presence of glycerol before transesterification was completed. Carmaker *et al* (2018) reported that acid esterification was unfavorable at high temperature (70 ℃) due to loss of methanol, reaction equilibrium also shifts away from product formation (34). Chand *et al* (2013) reported that temperature above the boiling point of alcohol was avoided because above 65 ℃ methanol evaporated. Hence biodiesel yield is lowered at high temperature (33). Figure 5 represents the biodiesel yield at different reaction temperatures.

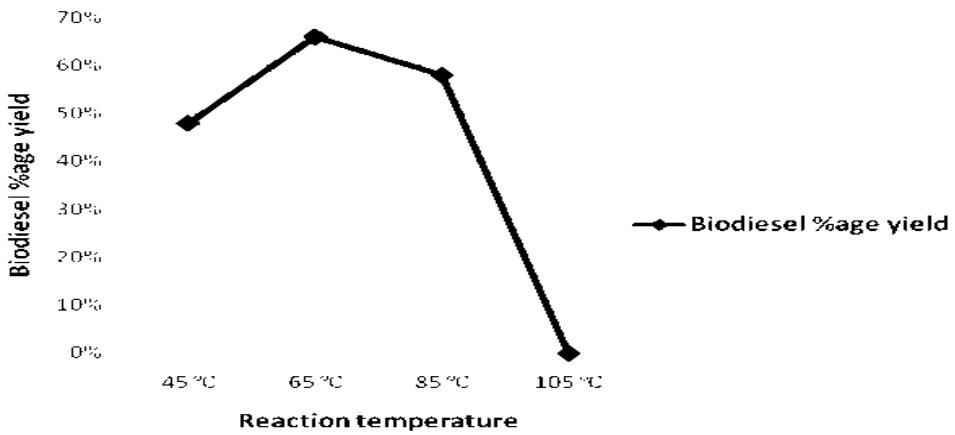


**Figure 5:** Effect of reaction temperature on biodiesel percentage yield.

**Effect of reaction time**

Four transesterification reactions were performed by using 5 mL of castor oil and variable reaction times such as 1 hour, 2 hours, 3 hours, and 4 hours. Methanol concentration, conc. $H_2SO_4$ catalyst concentration, reaction temperature, and stirring speed were 1.75 mL, 0.08 mL, 65 ℃ and 600 rpm, respectively. Obtained biodiesel yields were 0%, 28%, 42% and 0%. Phase separation was done when reaction times were 2 hours and 3 hours. But phase separation was not done at 1 hour and 4 hours of reaction time.

It was noticed that a maximum of 42% biodiesel yield was obtained at 3 hours of reaction time. But at the first hour of reaction time, we did not proceed with the transesterification process, because in this time it was below the limit of transesterification That is why phase separation was not done and the percentage yield was 0%. Phase separation was also not done at 4 hours because reaction time was over the limit for transesterification. Above 4 hours of reaction time, obtained biodiesel and obtained glycerol reversely start mixing with each other and spoil the biodiesel. That is why at 4 hours of reaction time biodiesel percentage yield was also 0% Finally, 3 hours of reaction time were considered as the optimal value for complete transesterification. Carmaker *et al* (2018) reported similar results that mass transfer between oil and alcohol is enhanced by adequate reaction time provided for reactants to interact (23). Silting *et al* (2016) and Seem *et al* (2015) are in agreement with our findings (35,36). Figure 6 represents the biodiesel yield at different reaction times.


**Figure 6:** Effect of reaction time on biodiesel percentage yield.

**Effect of stirring speed**

Four transesterification reactions were performed by using 5 mL of castor oil and variable stirring speeds such as 300 rpm, 400 rpm, 500 rpm and 600 rpm, respectively. Methanol concentration, conc. $H_2SO_4$ catalyst concentration, reaction temperature and reaction time were 1.75 mL, 0.08 mL, 65 ℃ and 3 hours, respectively. Obtained biodiesel yields were 22%, 38%, 42%, and 44%. Phase separation was also achieved for all transesterification reactions.

It was noticed that a maximum 44% biodiesel yield was obtained at 600 rpm of stirring speed due to proper mixing of oil and alcohol. But above 600 rpm yield was lowered because of improper mixing of oil and alcohol and chances of mixing up biodiesel and glycerol. So these changes resulted, that high stirring (600 rpm) being needed for the excitation of castor molecules for complete transesterification. Finally, 600 rpm was considered the optimal value for complete transesterification. Chand *et al* (2013) reported similar results that the highest yield was obtained at 600 rpm of stirring speed. Carmaker *et al* (2018), and Dhanani *et al* (2015) are in agreement with our findings (33,34). Figure 7 represents the biodiesel yield at different stirring speeds.

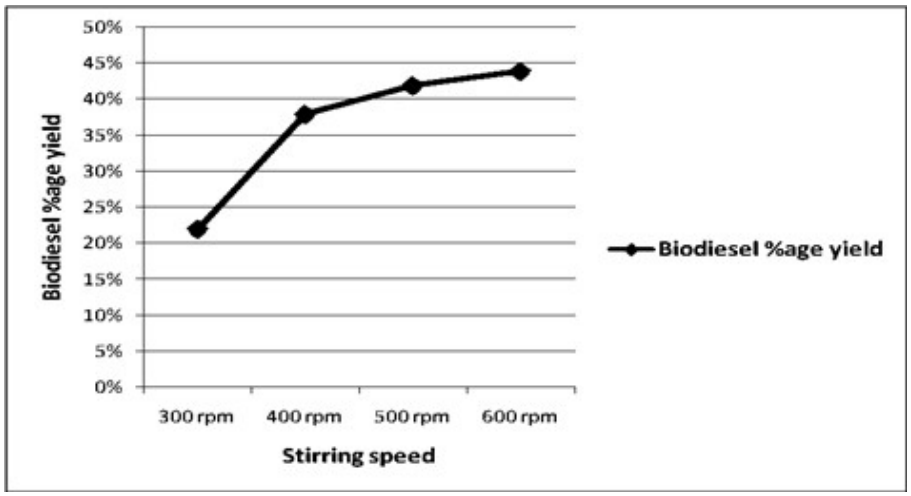


**Figure 7:** Effect of stirring speed on castor biodiesel percentage yield.

***Physicochemical properties of castor oil, castor biodiesel and blends***

Physicochemical properties of castor oil, castor biodiesel and blends $B_{10}$, $B_{20}$ & $B_{30}$ were measured. Blends were prepared on a volume-based ratio of biodiesel: diesel mixtures [$B_{10}$(1:9), $B_{20}$(2:8), $B_{30}$(3:7)]. Moreover, all measured values were also compared with standard tests. The data are given in Table 2.

**Table 2**: Characterization Summary of castor biodiesel.

| Properties | Units | Castor oil | Castor biodiesel | $B_{10}$ | $B_{20}$ | $B_{30}$ | Test method | Test limit |
|---|---|---|---|---|---|---|---|---|
| **Moisture contents** | percentage w/w | 0.171 | 0.112 | 1.441 | 1.4424 | 1.4397 | ASTM D2709 | 0.50 max |
| **Saponification value** | mg KOH/g | 186.35 | 86 | 1.43 | 2.36 | 4.32 | AOCS Cd3-25 | 175-187 |
| **Acid value** | mg KOH/g | 2.3002 | 1.0878 | 0.514 | 0.9635 | 0.9889 | ASTM D664 | 0.8 max |
| **FFA value** | mg KOH/g | 1.1501 | 0.439 | 0.757 | 0.9817 | 1.0939 | AOCSCa5 a40 | 3-38 |
| **Ester value** | mg KOH/g | 184.0498 | 84.9122 | 0.915 | 1.3965 | 3.3321 | EN14111 | 96.5 min |
| **Iodine value** | $gI_2$/100 g | 86.32 | 86.32 | 49.55 | 54.23 | 56.86 | AOCS Cd1-25 | 84-112 |
| **Peroxide value** | Me/100 g | 15.4 | 20.66 | 16.01 | 17.74 | 18.44 | - | - |
| **Specific gravity** | Gram | 0.959 | 0.8850 | 0.688 | 0.693 | 0.701 | ASTM D6751 | 0.87-0.90 |
| **Viscosity at 40 ℃** | cSt | 239 | 17.21 | 3.87 | 4.53 | 4.99 | ASTM D445 | 1.9-6 |
| **Refractive index** | nD | 1.4782 | 1.4667 | 1.441 | 1.4424 | 1.4397 | ASTM D960 | 1.32 |
| **Density** | Gram | 0.9481 | 0.8910 | 0.920 | 0.928 | 0.932 | ASTM D1298 | 0.83-0.89 |
| **Boiling point** | ℃ | 316 | 290 | - | - | - | - | - |
| **Molecular weight** | Gram | 914.42 | 1982.05 | - | - | - | - | - |
| **Higher heating value** | MJ/Kg | 40.494 | 44.479 | 48.628 | 48.519 | 48.399 | ASTM D240 | - |

**Moisture contents**

High moisture contents were present in castor oil (0.171%). Moisture contents of castor biodiesel were also high (0.112%). Khalil *et al* (2017) reported the moisture contents of castor oil as 4.4% (23). Keera *et al* (2018) reported the moisture contents of biodiesel as 0.02% (37). ASTM D2709 test limit of moisture content is 0.5 (max). Moisture contents percentage must be low in biodiesel because it promotes microbial growth in the biodiesel.

**Saponification value**

Saponification is the major problem of castor oil for biodiesel production. It is also affected on biodiesel yield. A basic catalyst like KOH and NaOH enhanced the saponification during transesterification. To overcome this problem acid catalyst was used for transesterification.

The saponification value of castor oil and its biodiesel were 186.35 mg KOH/g and 86.0 mg KOH/g. It decreased after transesterification. Saponification values of blends $B_{10}$, $B_{20}$ & $B_{30}$ were 1.43 mg KOH/g, 2.36 mg KOH/g, and 4.32 mg KOH/g, respectively. The saponification value of blends from $B_{10}$ to $B_{30}$ increased as the biodiesel contents in blends increased. Seem *et al* (2015) reported the saponification value of castor oil as 226.54 mg KOH/g (36). Bursary *et al* (2017) reported the saponification value of castor biodiesel as 241.55 mg KOH/g for base-catalyzed transesterification (38). AOCS Cd 3-15 test limit of saponification value for oil is 175-187 mg KOH/g. saponification value of castor oil is closed to AOCS Cd 3-15 test limit. Maximum reduction of saponification value from 186.35 mg KOH/g to 86.0 mg KOH/g took place for biodiesel which

resulted that better efficiency of biodiesel due to less specific matter. All blends showed a very low saponification value which means that they have better efficiency as a diesel substitute.

**Acid value**

The acid value is the number of a milligram of KOH required to neutralize the free fatty acids in 1 g of fats. A low acid value is favorable for biodiesel production**.** The acid value of castor oil was 2.3002 mg of KOH/g and after transesterification, it was decreased to 1.0878 mg of OH/g. Acid value of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 0.5147 mg of KOH/g, 0.9635 mg of KOH/g, and 0.9870 mg of KOH/g, respectively. The acid value of blends from B10 to B30 increased as the biodiesel contents in blends increased. ASTM D664 test limit of acid value is 0.8 mg of KOH/g (max). Chand *et al* (2013) reported the acid value of biodiesel as 0.8 mg KOH/g (33). Amite *et al* (2014) reported the acid value of castor oil as 2.629 mg KOH/g but they did not measure the acid value of castor biodiesel (15). But our measured acid value of castor biodiesel was 1.0878 mg KOH/g. All above reported values are close to our findings. All blends showed acid values within the range of the ASTM D664 test limit which means that blends can be used as diesel substitutes.

**Free fatty acid (FFA) value**

The high free fatty acid (FFA) value of castor oil tends to resist biodiesel production during transesterification. >2% free fatty acid value is an acceptable range for biodiesel production reported by Maher *et al* (2004) (19). Free fatty acid percentage of castor oil and its biodiesel were 1.1501 mg of KOH/g and 0.439 mg of KOH/g, respectively. FFA value of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 0.7573 mg of KOH/g, 0.9817 mg of KOH/g, and 1.0939 mg of KOH/g respectively. It was observed that the FFA value of blends from $B_{10}$ to $B_{30}$ increased as the biodiesel contents in blends increased. The standard the value of AOCS Ca5a-40 test limit for oil is 3.38-38.2 mg OH/g. Amite *et al* (2014) reported the FFA value of castor oil as 1.345 mg of KOH/g (15). All blends showed the minimum FFA value which means that blends can be used as diesel substitutes.

**Ester value**

Ester value is the number of mg of KOH required to specify the esters in 1 g of substance. The ester values of castor oil and its biodiesel were 184 mg of KOH/g and 84.2 mg of KOH/g, respectively. It decreased after transesterification. Ester value of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 0.9153 mg of KOH/g, 1.3965 mg of KOH/g, and 3.3321 mg of OH/g, respectively. It was observed that the ester value of blends from $B_{10}$ to $B_{30}$ increased as the biodiesel contents in blends increased. Seem *et al* (2015) reputed the ester value of castor oil as 218.47 mg of KOH/g (36). Stagey *et al* (2014) reported the ester value of castor biodiesel as 84.9122 which is similar to our findings (20). It was clear that a large number of ester contents may specify in castor oil than biodiesel. Whereas, all blends gave positive results of ester values.

**Iodine value**

The iodine values indicate the level of unsaturation in the oil. The fatty acid (ricinoleic acid, linoleic acid, oleic acid) present in castor oil makes the castor oil unsaturated. Unsaturation makes the fatty acid reactive, unstable and combustible with oxygen in the engine. The iodine test indicates the estimation of unsaturated compounds. More iodine consumption during tests means that high degree of unsaturation. The iodine value of castor oil and its biodiesel were 86.32 g of $I_2$/100 g oil. The iodine value of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 49.55 g of $I_2$/100 g oil, 54.23 g of $I_2$/100 g oil & 56.86 g of $I_2$/100 g oil respectively. Seem *et al* (2015) reported the iodine value of castor oil as 87 g of $I_2$/100 g oil (36). AOCD Cd1-25 test limit of iodine value is in the range of 84.2-112 g of $I_2$/100 g oil. It was clear that castor oil and its biodiesel have a high degree of unsaturation but blending the biodiesel with petro-diesel decreased the unsaturation.

**Peroxide value**

Peroxide value is used to determine the oil's oxidative rancidity. The peroxide value of castor oil and its biodiesel were 15.4 Meq/100 g and 20.66 Meq/100 g. it was increased after transesterification. Epoxide values of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 16.01 Meq/100 g, 17.74 Meq/100 g & 18.44 Meq/100 g respectively. It was observed that the peroxide value of blends from $B_{10}$ to $B_{30}$ was increased as biodiesel contents in blends increased. Stagey *et al* (2014) reported the peroxide value of castor and its biodiesel as 20 mg of KOH/g and 28 mg of KOH/g (39). It was clear that castor oil and its biodiesel have high oxidative rancidity due to high peroxide value, but blending positively lowers the peroxide value.

**Specific gravity**

The specific gravity of castor oil and its biodiesel were 0.959 g and 0.885 g, respectively. It decreased after transesterification. Specific gravity value of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 0.668 g, 0.693 g, and 0.701 g respectively. Specific gravity values of blends from $B_{10}$ to $B_{30}$ were increased as the biodiesel contents in blends increased. ASTM6751-02 test limit of specific gravity is 0.87-0.90 g. Maryam *et al* (2018) reported the specific gravity value of castor oil and castor biodiesel as 0.9 g and 0.5 g, respectively (10). Chand *et al* (2013) reported the specific gravity value of castor biodiesel as 0.961 g (33). Stagey *et al* (2014) reported the specific gravity value of $B_{10}$, $B_{20}$ and $B_{30}$ as 0.856 g, 0.860 g and 0.873 g respectively which are similar to our findings (39).

**Viscosity**

Viscosity is the measure of internal fluid resistance of oil to flow. High viscosity is not efficient for diesel. The observed viscosity value of castor oil was 239 cSt at 40℃. It was a high value. Transesterification was performed to minimize the viscosity value as 17.21 cSt at 40 ℃. Transesterification make the viscosity value in a favorable range. When biodiesel contents were blended with petro diesel then it was observed that the viscosity value was further decreased. Observed viscosity of blends such as $B_{10}$, $B_{20}$ & $B_{30}$ were 3.87 cSt, 4.53 cSt & 4.99 cSt respectively. ASTM D445 test limit of viscosity of biodiesel is 1.9-6.0 cSt. Stagey *et al* (2014) reported the viscosity value (at 40 ℃) of $B_{10}$, $B_{20}$ and $B_{30}$ as 3.78 cSt, 4.54 cSt, and 4.8 cSt (39). Deep *et al* (2017) reported the viscosity value (at 40 ℃) of castor oil and castor biodiesel as 240 cSt and 14.3 cSt, respectively (40). Maximum reduction of viscosity was observed when biodiesel is blended with petrodiesel. This resulted in that blends can be used as diesel substitutes.

**Refractive index**

The Refractive index value of castor oil and its biodiesel were 1.4782 nD and 1.4667 nD, respectively. Refractive index value of blends such as B10, B20 & $B_{30}$ were 1.4418 nD, 1.4424 nD, and 1.4397 nD, respectively. ASTM D960-79 test limit of refractive index is 1.32 (max). Maryam *et al* (2018) and Amite *et al* (2014) agree with our findings (10,15).

**Density**

The density of castor oil and its biodiesel were 0.9481 g/cm$^3$ and 0.8910 g/cm$^3$ respectively. Density value of blends such as $B_{10}$, $B_{20}$ and $B_{30}$ were 0.920 g/cm$^3$, 0.928 g/cm$^3$ and 0.932 g/cm$^3$ respectively. ASTM D1298 test limit of biodiesel is 0.830-0.890. Tonoi et al (2016) reported the density value of castor biodiesel as 0.880 g/cm$^3$ (21)**.** Stagey *et al* (2014) reported the density value of $B_{10}$, $B_{20}$ and $B_{30}$ as 0.856 g/cm$^3$, 0.860 g/cm$^3$ and 0.873 g/cm$^3$, respectively (39). All these values are close to our findings.

**Boiling point**

The boiling points of castor oil and its biodiesel were 316 ℃ and 290 ℃. All blend samples were evaporated at high temperatures.

**Average molecular weight (MW)**

The average molecular weight of castor oil and its biodiesel was 914.42 g and 1982.05 g, respectively. Rahman *et al* (2016) reported the average molecular weight of castor oil as 928 g (41).

**Higher heating value (HHV)**

A higher heating value is the amount of heating energy released by the combustion of a unit value of a fuel. The greater the higher heating value, the lower the fuel consumption. The higher heating value of castor oil and its biodiesel was observed as 40.4949 MJ/kg and 44.4792 MJ/kg respectively. The higher heating value of $B_{10}$, $B_{20}$ and $B_{30}$ were 48.6288 MJ/kg, 48.5198 MJ/kg and 48.3999 MJ/kg respectively. Silliman *et al* (2014) reported the higher heating value of castor biodiesel, B10, B20 and B30 were 38.576 MJ/kg, 41.626 MJ/kg, and 41.235 MJ/Kg and 40.810 MJ/Kg respectively (42)**.** Murat *et al* (2013) reported the higher heating value of castor biodiesel as 42.20 MJ/Kg which is close to our findings (43)**.**

**Instrumental characterization**

Functional groups of castor oil, castor biodiesel, and $B_{10}$, $B_{20}$ and $B_{30}$ were determined by using an FTIR spectrometer (see Figures 8a-8e).

Castor oil, castor biodiesel and blends such as $B_{20}$ and $B_{30}$ showed a broad band in the range of (3500 cm$^{-1}$- 3000 cm$^{-1}$) which indicated the (-OH) stretching vibration of alcohol, phenol, and carboxylic acid. $B_{10}$ did not show any band in the range of (3500 cm$^{-1}$ - 3000 cm$^{-1}$) which indicated the absence of the –OH group. The absorption of high intensity peaks of castor oil (2924.13 cm$^{-1}$, 2853.99 cm$^{-1}$), castor biodiesel (2924.60 cm$^{-1}$, 2854.31 cm$^{-1}$), $B_{20}$ (2924.83 cm$^{-1}$, 2854.43 cm$^{-1}$) and $B_{30}$ (2925.08 cm$^{-1}$, 2855.33 cm$^{-1}$) indicated the stretching vibration of –$CH_3$ group. All those samples having a frequency in the range of (3000 cm$^{-1}$ – 3500 cm$^{-1}$) show the presence of the –$CH_3$ group. $B_{10}$ was also indicated in the –$CH_3$ group but peak frequency was low. The strongest vibrations of castor oil (1742.60 cm$^{-1}$), castor biodiesel (1742.59 cm$^{-1}$), $B_{20}$ (1739.21 cm$^{-1}$) and $B_{30}$ (1740.81 cm$^{-1}$) were detected which indicated the C=O in carboxylic, ketone or aldehyde groups, suggested the presence of ester in all above samples. $B_{10}$ did not show any peak in this range which indicated the absence of the ester group. Another vibrations of castor oil (1458.65 cm$^{-1}$), castor biodiesel (1458.07 cm$^{-1}$), $B_{10}$ (1457.13 cm$^{-1}$). $B_{20}$ (1458.10 cm$^{-1}$) and $B_{30}$ (1458.92 cm$^{-1}$) indicated that the stretching vibration of -C-H (alkane) in mono, di and triglycerides glycerol in all samples. The peaks of all samples except $B_{10}$ in the range of (1245 cm$^{-1}$ – 1033 cm$^{-1}$) indicated the vibrations of (C-O) and (C-O-C) group, while an additional peak was observed at (1031.87 cm$^{-1}$) indicated the O-$CH_3$ stretching presence in the biodiesel spectrum (44). $B_{10}$ just showed the indication of the –$CH_3$ group and –C-H group. All this is because of a high volume of petro diesel hydrocarbons, the biodiesel character disappeared.

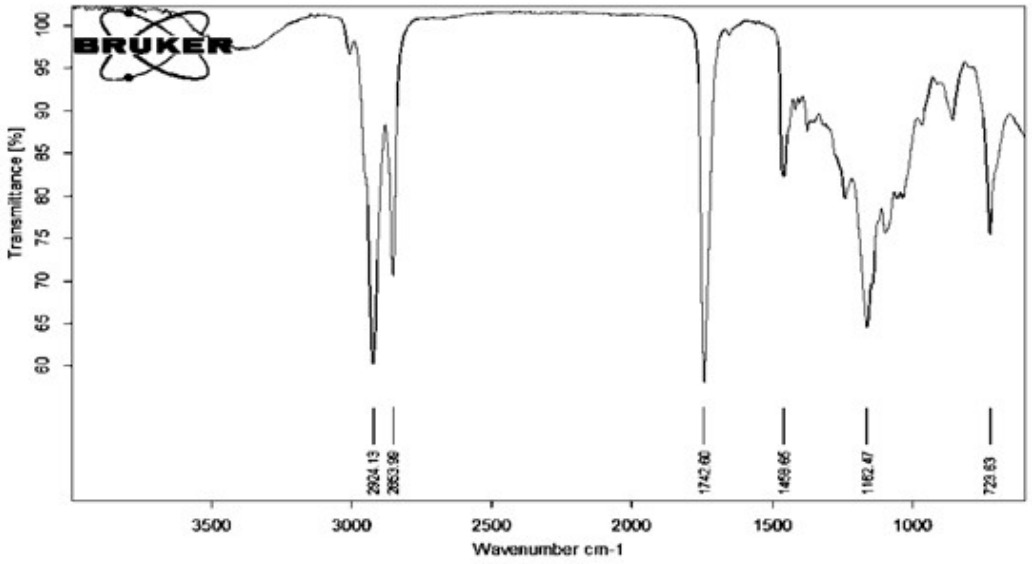


**Figure 8a:** FTIR spectrum of castor oil.

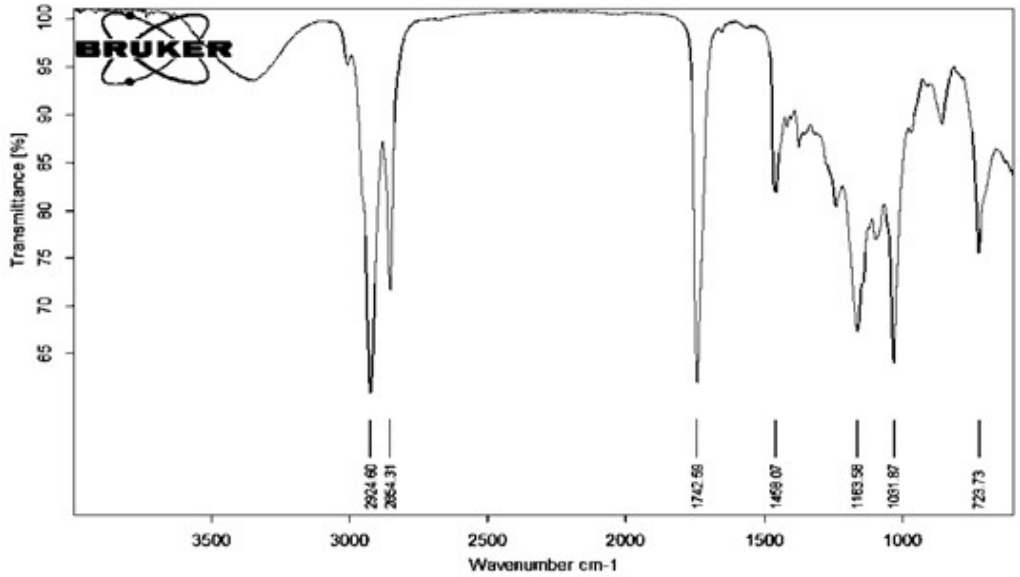


**Figure 8b:** FTIR spectrum of castor biodiesel.

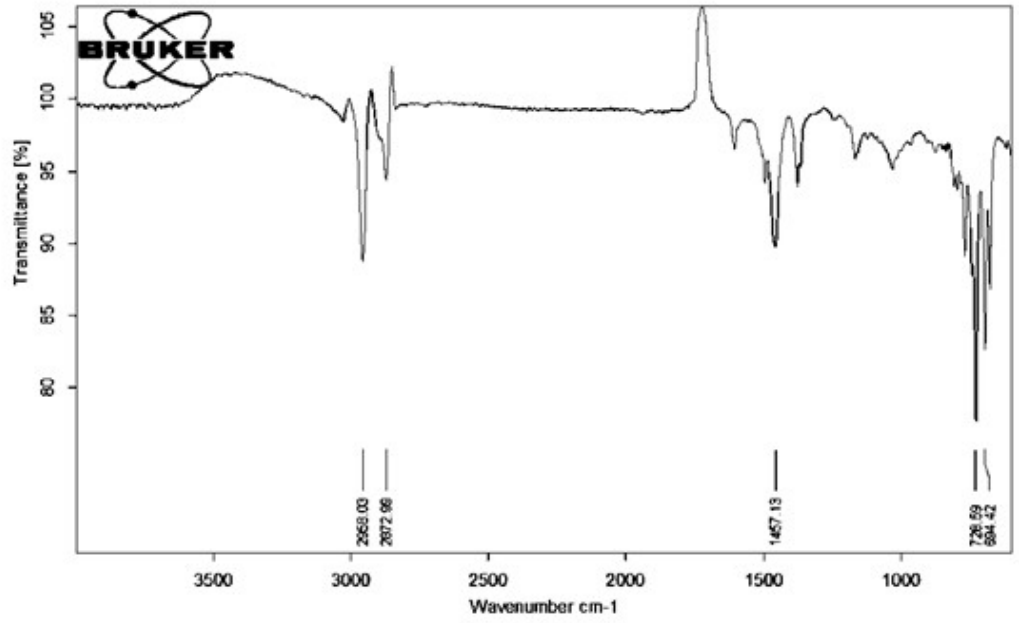


**Figure 8c:** FTIR spectrum of $B_{10}$.

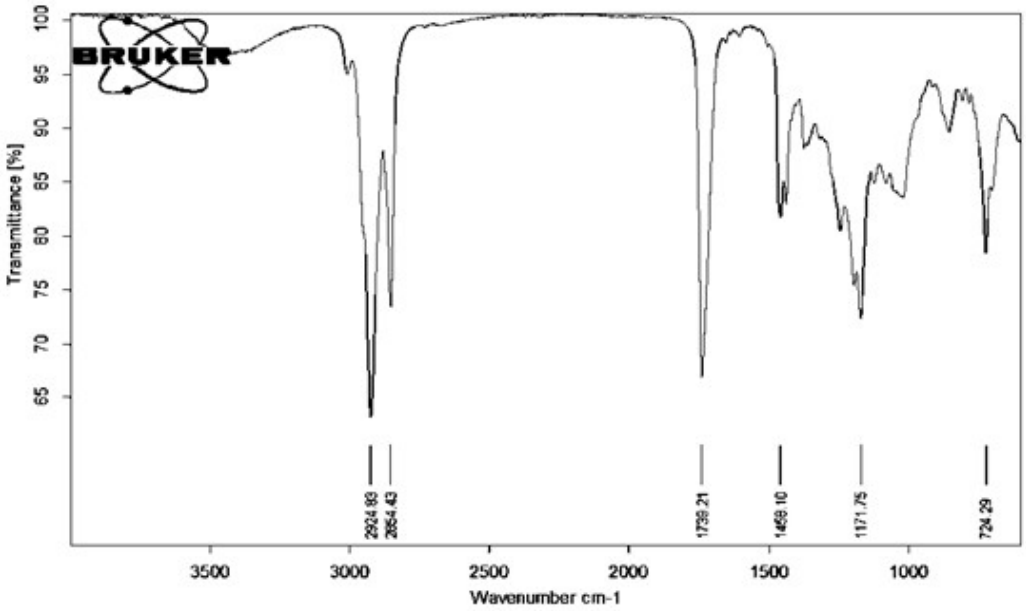


**Figure 8d:** FTIR spectrum of $B_{20}$.

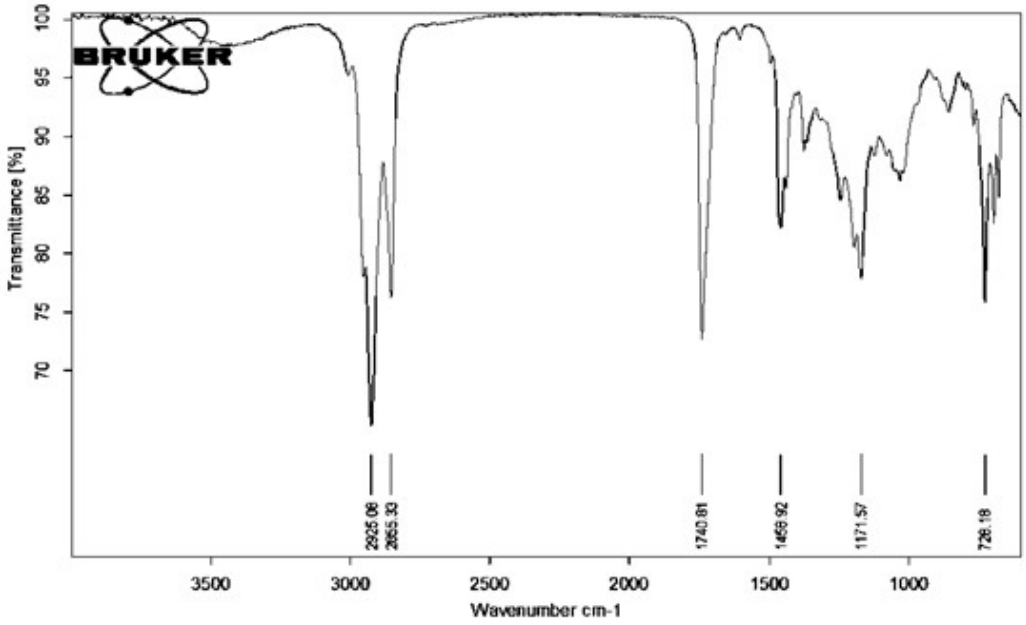


**Figure 8e:** FTIR spectrum of $B_{30}$.

## CONCLUSION

The overall research work based on the biodiesel production from castor oil, optimization of castor oil for obtaining a maximum yield of castor biodiesel, physico-chemical and instrumental characterization of castor oil and its blends were carried out. The maximum yield of castor biodiesel was 80% by using 20 mL of castor oil, 7 mL of methanol, 0.32 mL of conc. $H_2SO_4$ catalyst, 65 ℃ of reaction temperature, 3 hours of reaction time and 600 rpm of stirring speed. Moisture content (0.171 w/w to 0.112 w/w), saponification value (186.35 mg of KOH/g to 86 mg of KOH/g), acid value (2.3002 mg of KOH/g to 1.0878 mg of KOH/g), free fatty acid value (1.1501 mg of KOH/g to 0.439 mg of KOH/g), ester value (184.0498 mg of KOH/g), specific gravity (0.959 g to 0.8850 g), viscosity (239 cSt to 17.21 cSt), refractive index (1.4782 nD to 1.4667 nD), density (0.9481 g to 0.8910 g) and boiling point (316 °C to 290 °C) decreased after transesterification. The iodine value of castor oil and its biodiesel remains the same (86.32 g of $I_2$/100 g). Peroxide value (15.4 meq/100 g to 20.66 meq/100 g), molecular weight (914.42 g to 1982.05 g) and higher heating value (40.494 MJ/kg to 44.479 MJ/kg) increased after esterification. From FTIR spectra, it is investigated that all samples contain an ester group except $B_{10}$. Actually, the high volume of petro-diesel in the blend $B_{10}$, Ester group disappeared but petro diesel properties enhanced. Finally blend samples $B_{20}$ and $B_{30}$ were recommended as diesel substitutes because the physicochemical properties of these samples were in favorable ranges. $B_{10}$ showed a similar character to petro diesel due to the high volume of petro diesel the biodiesel character disappeared.

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## DISCLOSURE STATEMENT

No potential conflict of interest was reported by the authors.

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