

# *In situ* Transesterification/Reactive Extraction of Castor Bean Seeds Assisted by Flying Jet Plasma for Biodiesel Production

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## Abstract

One of the most exciting areas for the development of alternative fuels is the production of biodiesel. To reduce the cost of biodiesel production, *in situ* trans-esterification has been introduced to simplify the production process by enabling extraction and trans-esterification to occur at a single stage in the presence of a catalyst. In this study, we investigated the feasibility of using non-corrosive and environmentally receptive flying jet plasma as an alternative catalytic route for *in situ* tran-esterification of castor bean seeds (CBS). Upon optimizing the reaction conditions, it is elucidated that applying a low ratio of methanol to seeds ( $\leq 6:1$ ) has resulted in hindering the *in situ* trans-esterification and leading to insignificant conversion. The yield of esters has increased from 80.5% to 91.7% as the molar ratio rose from 9:1 to 12:1. Excess alcohol beyond the ratio of 15:1 was shown to have a negative impact on the yield of the produced esters, attributed to an increase in the biodiesel portion prone to dissolving in the co-product (glycerol). An increase in the reaction bulk temperature from 40 to 55 °C led to a higher ester content by 50%. Further increases in the bulk temperature beyond 55 °C did not affect yields. Regarding the reaction period, the results have shown that 3 h of reaction is adequate for a higher biodiesel yield. The quality of the biodiesel obtained has demonstrated that all physicochemical properties meet the ASTM D6751 specifications.

**Keywords:** *Flying jet plasma, In situ transesterification, Reaction mechanism, Biodiesel, Chromatographic analyses*

## 1. Introduction

Biodiesel is an alternative diesel fuel consisting of alkyl monoesters of fatty acids from vegetable oils or animal fats. In the past few years, numerous catalysts have been extensively studied to improve production quality. It is worth mentioning that alkali catalysts have exhibited superior performance, achieving more than 90% biodiesel yield within less than 60 min at a reaction bulk temperature of less than 60 °C.

The restraint of using alkali catalysts is attributed to high sensitivity towards the presence of high free fatty acids (FFA) in low-cost feedstocks[1]. Prior to forming the triglycerides, FFA in high concentration consumes the alkali catalyst, leading to soap formation, which is considered the most challenging, along with the removal of the remaining catalyst in the biodiesel purification process[2]. To resolve this problem, several researchers have suggested the use of an acid catalyzed process. These catalysts were found to be slower, necessitating higher reaction temperatures ( $> 100$  °C) and more purification steps[3].

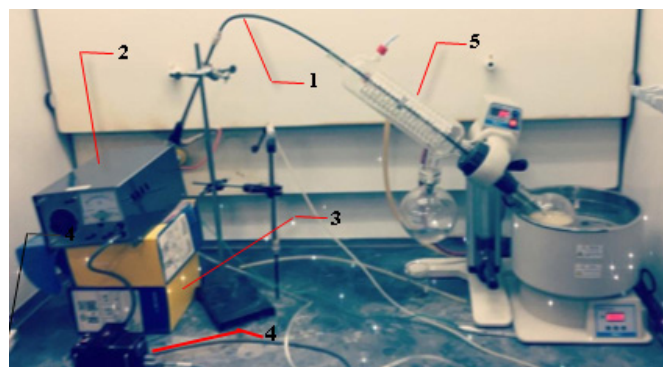
Recently, several studies have investigated the application of different

heterogeneous catalysts in biodiesel preparation as their removal is easy. Even with remarkable heterogeneous catalysis advantages (e.g., product purity and yield), most heterogeneous catalysts are deactivated within a short period due to leaching of catalyst active species, glycerol recovery, and the ability to catalyze the feedstocks containing high FFA[4].

Newly, the use of plasma catalysis has gained considerable attention from numerous research groups as it seems to be promising in catalysis[5-6,8-10,24]. Jet plasma, a category of non-thermal plasma, consists of a host of high-energy electrons, free radicals, chemically active ions and attracted species; and is therefore applicable as a unique catalyzing agent for a broad spectrum of chemical processes. Compared to others, no moving parts, lower mass and fast response make the jet plasma superior for any application[6].

The main objective of this research is to replace the traditional catalytic systems with new catalytic tools/materials capable of solving the drawbacks of transesterification catalysts, improving the reaction progress, thereby simplifying the purification and reducing the production cost. It is not worthy that the stumbling block of biodiesel is the high production cost imputed to the costly vegetable oil refining processes. The refining process cost was estimated to be 70-95% of the total biodiesel production cost[1,7]. In this research, an alternative low-cost feedstock CBS is examined for direct conversion into biodiesel through one shot *in situ* transesterification with methanol, overstepping the extraction and purification steps.

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1. Hollow plastic tube equipped with copper wire.
2. Plasma power source.
3. Platform.
4. Transformer.
5. Condenser.



6. Jet plasma plume inside the liquid bulk.
7. Rotating glass bowl.
8. Hollow plastic tube equipped with copper wire.

**Figure 1.** The FJPT reaction system.

## 2. Materials and methods

### 2.1. Materials

The castor seeds were collected from WadiFanja, Al Dakhiliyah Governorate, Oman. Methanol ( $\geq 99.8\%$  purity) purchased from Sigma Aldrich was applied as an alcoholic agent in the transesterification reaction.

A considerable part of this work was devoted to studying the role of non-thermal plasma as an emerged catalyzing agent. Custom made reaction system based on integrating a flying jet plasma torch with a rotary evaporator was used in this research, Figure 1, as in the following details:

#### 2.1.1. Flying Jet Plasma Torch (FJPT)

This research used a developed dielectric barrier discharge platform –FJPT as a sole agent to catalyze CBS transesterification. The torch was powered by a plasma power source (DIDRIV10)[8-9], purchased from Information Unlimited/USA. The device is equipped with independent voltage control from zero to a maximum output of 40 kV, independent current control from 5% to maximum output and independent frequency control from 20 to 70 kHz. Argon gas of purity 99.995%, purchased from Muscat Gases Co., was applied through a Teflon flow meter (Tucsen/ China) at a rate of 100 ml/min for the jet plasma generation.

The generated plasma was characterized by plotting the voltage – charge diagram (Lissajous figure)[10,11] using a high voltage capacitor (200  $\mu$ F) connected in series with the load. The generated plot estimates the energy consumed by the discharge per cycle of the applied voltage, in which the mean dissipated power is deduced from the applied frequency and the estimated energy ( $P = f \times E$ ).

#### 2.1.2. Rotary evaporator

A diagonal digital rotary evaporator WEV-1001L, purchased from Daihan/Korea, was used as a reaction bulk. This facility ensured con-

trolled reaction conditions in terms of temperature and rotation speed. The device could be operated in a range from 0 to 99 °C and from 10 to 180 rpm).

### 2.2. CBS *in situ* transesterification procedure

Before starting the *in situ* transesterification experiments (reaction Scheme 1), the amount of seeds, the content of fats, and the amount of required alcohol were estimated from the stoichiometric ratios of the balanced equation. Hence, oil was extracted from the crushed CBS using n-hexane as solvent via the modified Soxhlet apparatus. From trial tests, seeds were found to be potentially competitive in terms of oil content (around 40 wt%). Subsequently, the mean molecular weight ( $M_w$ ) was calculated using the formula in (Eq. 1) based on its saponification value ( $SV$ )[12].

$$M_w = \frac{56,000}{SV} - 12.67 \quad (1)$$

In order to calculate the reaction stoichiometry, the quantity of seeds required for the reaction was determined from the oil quantity obtained in the Soxhlet multiplied by the oil percentage (Eq. 2).

$$Seeds(g) = \frac{Extracted\ amount\ of\ oil(g)}{oil\%} \quad (2)$$

In this work, *in situ* transesterification was conducted in a reactor shown in Figure 1, in which the mixtures were induced under reflux condensation. The rotary evaporator is a part of this design as it provides condensation of methanol, rotation to maintain uniform mixing during the experiment, and a slot for inserting the plasma feeder wire.

The plasma-catalyzed *in situ* transesterification reaction was assessed through several parameters: methanol to seeds molar ratios, reaction temperature, and the reaction time (range up to 4 h). A fixed rotation speed of 35 rpm for the reaction bulk was set to overcome any poten-

tial mass transfer diffusion limitation throughout the process.

At the proposed conditions, the reactants, the methanol, and the crushed seeds were induced with plasma in a digital rotary evaporator, as illustrated above in Figure 1. During the reaction time, thin layer chromatography (TLC) was used to monitor the disappearance of the limiting reactant triglycerides (TG), the appearance and disappearance of the intermediate products, monoglycerides (MG) and diglycerides (DG), and the appearance of the produced methyl ester (ME). Two samples were taken for analysis in each 30 min to ensure the completion of the reaction. One was qualitatively analyzed by TLC to follow the reaction progress, and the other was quantitatively tested by gas chromatography (GC) to pursue the product revolution.

When the reaction reached the exposed time, the plasma device was switched off, and the excess methanol was gently collected in the rotary flask after increasing the temperature to 70 °C. Then, the solution mixture, which was supposed to contain the product and the derived by-product, was filtered from the transesterified seeds through filter paper.

The filtration step was achieved on a hot plate to ensure complete maceration of the filter paper. The filtrate was then entered into a separatory funnel, in which a spontaneous separation of two phases occurred after four hours without the need for any solvent being introduced. Finally, the phase separation was carried out by decanting the lower layer of glycerol easily from the upper layer of the biodiesel product.

### 2.3. Chromatographic analysis of CBS oil and methyl esters

The composition of CBS fatty acids and methyl esters was dictated by using the official AOCS method Ce 2-66 by using Agilent gas chromatogram (USA) Hewlett-Packard 6890 series outfitted with a flame ionization detector and the capillary column SP-2340 with a split ratio of 100:1 having a length of 60 m, an inner diameter of 25 mm and a film thickness of 0.2 µm.

The samples were diluted 100 times in n-hexane prior to injection. The oven temperature ramp program holds an initial temperature of

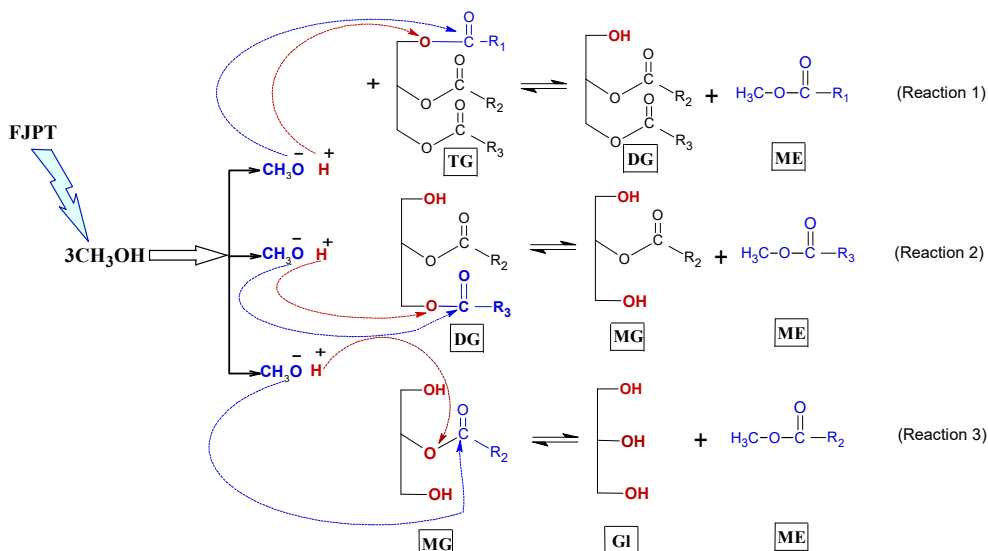
150 °C for 1.5 mins, then soared to 210 °C at a rate of 3 °C/min. The oven was kept for 30 seconds at that temperature, then rose to 240 °C at the same rate and retained at 240 °C for 8 min. Also, both detector and injector were kept at 240 °C. The carrier gas used was high-purity helium (> 99.95 mol%) streamlined at a flow rate of 0.8 ml/min. All the percentages of fatty acids were estimated in triplicate runs medium.

For trial experiments, the content of methylesters formed in the reaction was calculated from the actual content of esters achieved from GC to the theoretical yield calculated from reaction stoichiometry. All the data acquired are average results of triplicate determinations in each case. In addition to the GC analysis, a qualitative study of the presence of ME, MG, DG and TG in the samples was performed on a thin layer chromatography (TLC) plate. TLC study elucidates the revolution of the reactants and the evolution of the products within the reaction time. The extraction was achieved with a hexane-diethyl ether mixture of 9:1 (v/v)[13]. The samples were spotted on the TLC silica plate after they had been diluted in ethyl acetate. The component spots were shown as dark brown spots when the TLC plate was kept in the iodine chamber.

### 2.4. FJPT *in situ* transesterification mechanism

As indicated above, there are three sequential reversible reactions leading to transesterification. Generally, in each stage, a singular mole methyl ester (ME) together with MG, DG and GI is formed through the first, second and third paths, respectively (Scheme 1).

It is worth mentioning that the jet plasma encourages ionization inside the liquid bulk caused by the rapid development of the individual micro-discharge filaments[14]. Furthermore, since the target of using the plasma system is To dissociate methanol into proton hydrogen (H<sup>+</sup>) and methoxide anions (CH<sub>3</sub>O<sup>-</sup>), it is thus expected that such interactions between anions and cations would be significant to catalyze the transesterification reaction. Consequently, the filament diffuses out inside the liquid bulk. This may facilitate the movement of H<sup>+</sup> from the plasma interaction surface, leading to a nucleophilic attack on carbonyl carbon.



Scheme 1. Proposed mechanism for FJPT-catalyzed *in situ* transesterification reaction of CBS.

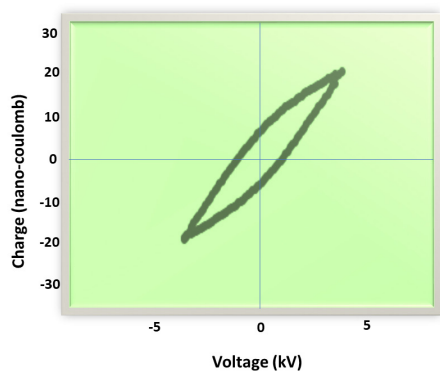


Figure 2. Lissajous figure generated for the DBD platform for one cycle when using argon gas and applying 30 kHz.

From the proposed reaction mechanism scheme, it can be seen clearly that the excess  $\text{CH}_3\text{O}^-$  has targeted the TG carbonyl groups in three positions. In the first step (Reaction 1), one of the TG molecule carbonyl carbon atoms attracts  $\text{CH}_3\text{O}^-$  and  $\text{H}^+$  to form ME and a tetrahedral intermediate of DG, respectively. In the second step, the DG interacts again with  $\text{CH}_3\text{O}^-$  and  $\text{H}^+$  to form ME and tetrahedral intermediate of MG (Reaction 2). Refers to the last step illustrated in Reaction 3, DG tetrahedral intermediate loses a carbonyl group and accepts hydrogen cation ( $\text{H}^+$ ), resulting in ME and GI formation.

### 3. Results and discussion

#### 3.1. Characterization of the generated jet plasma and its imposing duration

It is noteworthy that because of the existing lag phase between the voltage and the current in an alternating current power source, the consumption of electrical power in the plasma bulk does not firmly correspond to the total power drawn from the mains. Hence, an approximation to estimate the power consumed in the plasma bulk is commonly completed through generating the Lissajous figure (the voltage charge diagram)[10,11], in which the area confined by the lines corresponds to the energy consumed per cycle of the applied voltage. Hence, the mean dissipated power in the plasma bulk is interpreted through the product of this energy times the applied frequency.

In the current study, an optimized theoretical electrical power of 175 watts was induced in the plasma system (based on the root mean squared values of 2.5 kV and approximately 0.07 Amp measured close to the load, at 30 kHz). Thus, a uniform and stable plasma generation was maintained at a 100 mL/min argon flow rate, attaining a 0.5 cm plume length.

The charge-voltage diagram, shown in Figure 2, was generated for the DBD platform responsible for elaborating the jet plasma in this study. The plot resembles an Almond-shaped diagram and is attributed to the gradual expansion of the plasma across the available dielectric area during the discharge phase. Hence the confined area between the lines of the Lissajous diagram, Figure 2, was calculated (using Origin software) to be 4.5 mJ. Accordingly, the effective power delivered to the DBD cell was found to be around 135 watts. It can be noticed that

Table 1. Fatty Acid Composition of Castor Bean Seeds by Gas Chromatography Analysis

Peak no.	Ret. Time	Acid name	Abbreviated Formula	Proportion (%)
1	21.114	Palmitic	C16:0	14.53935
2	25.023	Stearic	C18:0	9.94073
3	26.259	Oleic	C18:1	31.10799
4	28.173	Linoleic	C18:2	37.109
5	30.388	Linolenic	C18:3	4.27941
6	30.597	Gadoleic	C20:1	2.31873

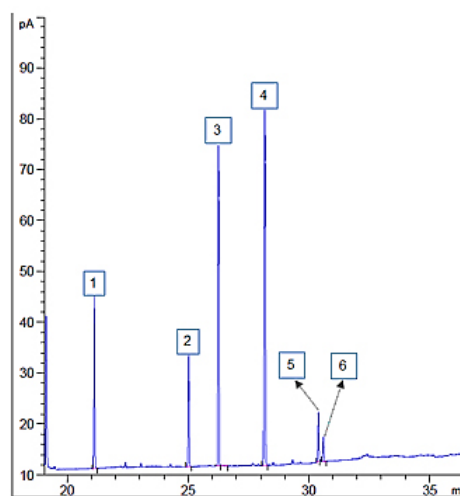


Figure 3. GC analysis of CBS fatty acids content.

only 77% of the source power is consumed in the plasma system; this is commonly attributed to losses in the power source circuit, transferring wires and the boundary. However, the aforementioned set of values for the power parameters was found optimal when compared to other examined sets; and hence it was adopted for the rest of the work in this study. These conditions permit a current density of  $8.9 \times 10^4$  Amp.m<sup>-2</sup> (based on inducing 0.07 Amp through 1 mm diameter copper wire of  $7.85 \times 10^{-7}$  m<sup>2</sup> cross-sectional area).

#### 3.2. Characterization of oil fatty acid content

The oil was extracted using the Soxhlet extraction method. Approximately 60 g of dried seeds were crushed and loaded into the Soxhlet extractor using n-hexane as solvent. In the extractor, the samples were heated to the solvent boiling temperature for 7 h. The solvent was then evaporated using a rotary evaporator. At 70 °C for 1 h, the extracted oil was dried in an oven before subjecting to gas chromatography coupled with flame ionization detection (GC-FID).

Table 1 and Figure 3 present the analysis of GC on CBS oil. The highest contents of fatty acids observed in the CBS oil are linoleic acid and oleic acid accounting for 68.2% of the total fatty acids. Many minor constituents, such as palmitic, stearic, linolenic, and gadoleic were also found in the oil.

### 3.3. Jet plasma-catalyzed *in situ* transesterification

#### 3.3.1. The effect of the methanol to seeds molar ratio on ME conversion

The molar ratio of alcohol to oil is among the most significant factors influencing conversion efficiency, yield, and production costs of biodiesel[15].

The effect of methanol to seeds ratio is examined in the designed plasma-assisted batch reaction mode under atmospheric pressure by differing their ratio, whereas the reaction temperature and agitation rate were fixed at 55 °C and 35 rpm, respectively. Six specific molar ratios of 3:1, 6:1, 9:1, 12:1, 15:1, and 18:1 were used on an experimental scale. It can be seen from Figure 4 that molar ratios of 3:1 and 6:1 resulted in lower yields of esters. In this process, methanol served as an extraction solvent as well as its role as a second reactant. As the reaction is reversible, a higher amount of alcohol was needed to increase the rate of the forward reaction and to improve the miscibility between the reactants (methanol and triglycerides) molecules. Another part of methanol is likely engaged in the extraction process[16]. In this sense, our findings clearly revealed that the molar ratios less than 6:1 is hindering the *in situ* transesterification process and led to poor productivity.

On the other side, ester's yield was increased from 80.5% to 91.7% as the molar ratio (methanol/seeds) increased from 9:1 to 12:1. Further increases in the molar ratio, i.e., 12:1 to 15:1, did not result in significant improvement in the derived esters yield. However, excess alcohol showed a negative impact on the esters yield when the molar ratio exceeded 15:1. This was attributed to the role of excessive alcohol, which created difficulties in separating biodiesel from the by-product (glycerol) as it complicated the phase separation and resulted in a portion of biodiesel left in the glycerol phase[15,16]. In addition, the alcoholic portion remained in the biodiesel product could influence the quality of the biodiesel fuel in terms of reductions in its viscosity, density and flash point[17]. This result was in good agreement with the finding of the earlier study by Sadrameli and Omare[18], who reported that the canola oil esters yield decreased when molar ratio of greater than 15:1 was employed.

#### 3.3.2. The effect of temperature on esters content

*In situ* transesterification, also referred to as reactive extraction, was implemented in this study to facilitate the process of production of biodiesel by allowing extraction and (trans)esterification to take place in a single stage. Temperature has been reported to have a major impact on the reaction/extraction single stage[19,20]. Therefore, five stages have been carried out to study the impact of temperature on esters production. Depending on the result of the previous part, the optimized 12:1 molar ratio was applied, whilst the conditions of rotation speed were fixed at 35 rpm for 3 h reaction duration under atmospheric pressure. Figure 5 illustrates the produced esters content from reactions conducted at 40, 45, 50, 55 and 60 °C. This research was conducted at low temperatures, as it is believed to be a cost-effective technique. It can also be observed that the lowest temperature in the applied range (40 °C) did not have any significant impact on the produced esters.

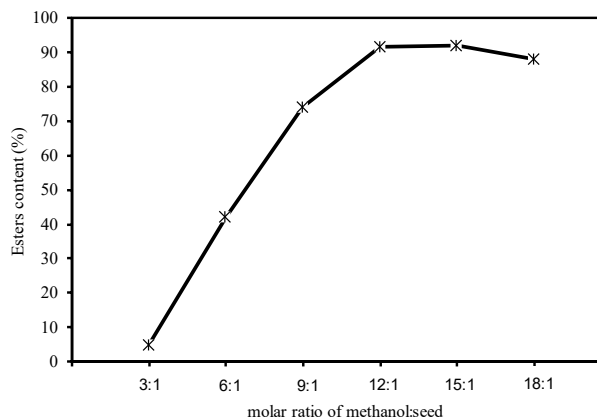


Figure 4. Effect of molar ratio of reactants on ester content.

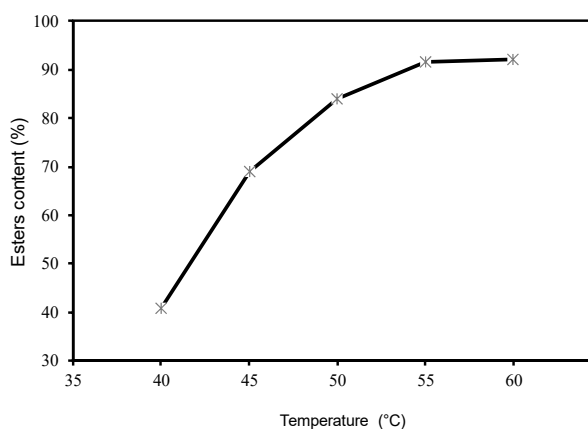


Figure 5. Effect of temperature on ester content.

However, an increase in the reaction bulk temperature from 40 to 55 °C raised the esters content gradually from 40.8% to 91.7%. This indicates that higher temperature bulk resulted in a positive impact on the efficiency of both extraction and reaction. Further increase in the reaction bulk temperature up to 60 °C did not show any advantages on the ester yield. It should be noted that many researchers have indicated that the best reaction bulk temperature is around 60 °C. This is applicable when using either alkali or acid catalysts[4]. This finding is consistent with the previous study conducted by Haas *et al.*[21], in which a temperature of 55 °C was introduced to achieve the highest yield of biodiesel (>90%) from *in situ* transesterification of distillers dried grains with soluble feedstock. In addition, Mehta *et al.*[22] demonstrated that plasma could bypass thermodynamic limitations to enable different reactions at low temperatures.

#### 3.3.3. The effect of reaction time on esters content

The transesterification process completion relies on the time of the reaction[23]. Hence, experiments were performed in several durations (30, 60–240 min) with the aforementioned optimized conditions (12:1 molar ratio and 55 °C) at a fixed rotation of 35 rpm.

After 30 min, as it can be seen in Figure 6, the limiting reactant TG emergently appeared in the TLC plate, indicating that the extraction

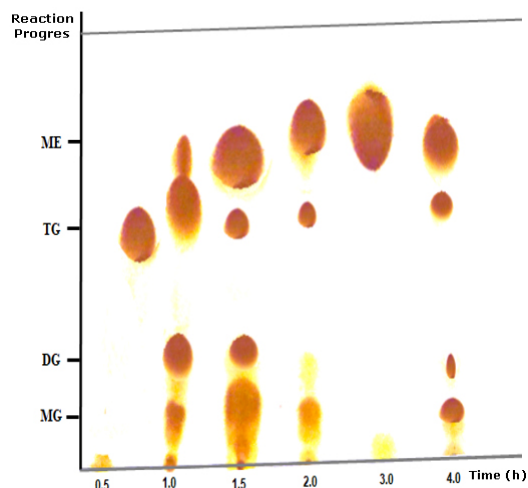


Figure 6. TLC result for reaction progress during the reaction time.

process had begun. However, no presence of the intermediate products (DG and MG) was observed, and as a result, no spots of ME were formed. It can be concluded that the transesterification reaction was initiated after 30 min of the start time since the ME along with MG and TG spots were observed in the plate. The experiment showed that ME content slowly increased from 34.5% to 76% during 60 min of the reaction time.

The highest yield was observed when approaching 3 h of the reaction start, in which the TG and other intermediates completely disappeared. The findings obtained have been supported by the GC analysis, illustrated in Figure 7. As it can be observed, the ester content improved upon increasing the reaction time. Hence, optimum ester content (91.7%) was obtained at 3 h of reaction start. Further increase in the reaction time, beyond 3 h, did not enhance the esters productivity, as illustrated in figures 4 and 5. Furthermore, it can be observed in Figure 6 that spots of MG and TG started to reappear. This could be attributed to the fact that transesterification encourages reverse reaction and tends to restore its equilibrium conditions[23].

### 3.4. Properties of the produced biodiesel

To be used as automobile fuel, the properties of the produced biodiesel must meet the specifications. Table 2 presented experimental data for density, boiling point, flash point, cloud point, saponification value, acid value, and sulphated ash for the produced biodiesel.

The standard methods ASTM D 445, ASTM D4052, ASTM D7398, ASTM D93, ASTM D2500, ASTM D5558, ASTM D664, ASTM D4530, and ASTM D2709 were used to determine the kinematic viscosity, density, boiling point, flash point, cloud point, saponification value, acid value, and sulphated ash for the produced biodiesel, respectively. As can be seen in Table 2, all the tested parameters of the produced Castor Bean biodiesel were found to be within the limits of ASTM D6751.

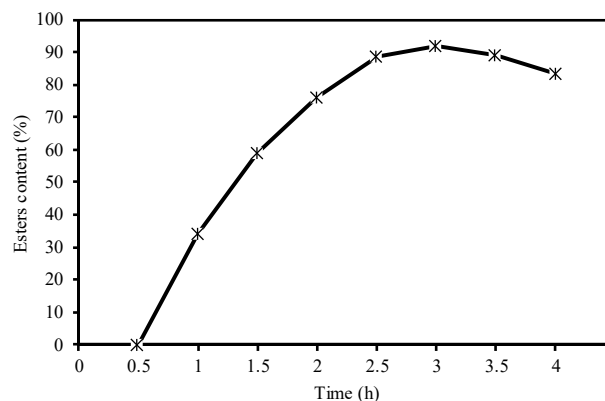


Figure 7. Effect of reaction time on ester content formation.

Table 2. Properties of Biodiesel Produced from CBS

Properties	Unit	Limits	Produced biodiesel
Kinematic viscosity (at 40 °C)	cSt	1.9-6.0	3.8
Density (at 15 °C)	kg/m <sup>3</sup>	-	844
Boiling point	° C	100-600	236
Flash point	° C	130 (min)	206
Cloud point	° C	Report	6
Saponification value	mg KOH/g	350 (max)	129
Acid value	mg KOH/g oil	0.5 (max)	0.35
Sulphated ash	wt%	0.02 max	0.0003
Water & Sediment	% vol	0.05 max	0.009

## 4. Conclusions

The current study demonstrated that biodiesel at high conversion could be produced with the aid of jet plasma *in situ* CBS transesterification process. Our results revealed that the temperature and the molar ratio (methanol/oil) have strongly influenced the conversion to ME. Optimized parameters of 55 °C, 12:1 molar ratio of alcohol to oil, and reaction time of 3 h were found adequate to complete the *in situ* transesterification and yielded more than 90% esters. More importantly, the produced methyl ester physicochemical properties fulfilled the international biodiesel standard suggesting a feasible approach to be adopted on an industrial scale.

### Data availability statement

The authors of this article confirm that no data, models, or code were generated or used during the study.

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