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# Improving Biodiesel Quality and Productivity from Egyptian Castor by Studying Optimization Factors

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#### Abstract

A main reason for Air pollution and greenhouse gas emissions is petroleum-based fuel, burning those types of fuels produces carbon monoxide, carbon dioxide which harms both environment & human's health, in addition to the great drop in petroleum sources' quantities recently and the continuous increase in its price leading to products raise in price, all that opened the door Biodiesel has shown a great performance in for new generation of fuel derived from natural origins such as biofuels. combustion engines as a clean alternative to petroleum-based diesel. Biodiesel can be derived from plant-based oil, or animal which is considered as a waste in Egypt and as an oil rich source is considered as a valuable source fat-based oil. Castor seed for bio diesel in Egypt. This study motivated enhancing the efficiency of biodiesel generation from castor oil by employing Response Surface Methodology. Three variables were studied: temperature, reaction time, and methanol concentration. The optimization aims to identify the optimal conditions to maximize biodiesel production with effective conversion efficiency. The optimization revealed that maintaining a temperature of 40 °C for 60 minutes, with a 1% KOH catalyst and 20% methanol by weight, resulted in the highest yield of biodiesel at 90.859% and a conversion efficiency of 82.04%. Quality assessment through thin-layer chromatography and physical property analysis of the refined methyl ester demonstrated compliance with or superiority over ASTM biodiesel standards.

Keywords: Biodiesel, Transesterification, Conversion, Biodiesel quality, Castor oil, Response Surface Methodology, Optimization.

#### 1. Introduction

Fossil fuels currently dominate the world's energy supply, but a global push for renewable alternatives is underway for two key reasons. Firstly, concerns are rising about the ever-increasing cost and unpredictable nature of fossil fuels. Secondly, there are growing environmental worries linked to fossil fuel use, such as air pollution and global warming [1][2]. Also, energy demand is on the rise globally, with fossil fuels like oil, natural gas, and coal accounting for roughly 80% of total consumption [3]. In this context, biofuels emerge as a promising solution. Being renewable and environmentally friendly, they can help bridge the gap in the world's energy needs [4][5].

Egypt, a vast country with roughly 1 million square kilometers and a population of 92 million (according to 2016 data), has seen a significant rise in diesel demand. Statistics show a 30% increase between 2005 and 2015. However, diesel prices also climbed sharply during this period, rising by 64% to \$230 per

ton (roughly 1.9 Egyptian pounds per liter) between October 2008 and July 2014. Notably, there aren't currently any policies in place to mandate a minimum 1% biodiesel blend with traditional diesel fuel [6] [7], [8].

A major concern driving research into alternative energy sources is the two-fold issue of dwindling fossil fuel reserves and their environmental damage [9]. Biofuels, like those derived from oilseeds or food waste, offer a renewable alternative. Many researchers believe biofuels hold promise because they can lessen greenhouse gas emissions and air pollution, while also reducing reliance on imported fuels and potentially lowering overall energy costs. Essentially, plant-based fuels are seen as a key alternative to fossil fuels [10]. Importantly, these biofuels, often called biodiesel, can be used in existing diesel engines [3],[11]. Biodiesel, meeting ASTM standards, consists mainly of mono-alkyl esters sourced sustainably from vegetable oils or animal fats meeting ASTM D6751 requirements [9].

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It offers environmental advantages through reduced greenhouse gas emissions, being renewable, nonbiodegradable, and lacking compounds. Castor oil biodiesel, for example, has a lower cetane number than regular diesel [12][13][14]. Notably, biodiesel's high oxygen content aids in ignition in diesel engines and is sulfur-free, preventing harmful sulfur oxide emissions [15] . Glycerides and vegetable oils' high calorific value makes them excellent for biodiesel production. Using unrefined vegetable oils can further lower greenhouse gas emissions by absorbing carbon dioxide during crop growth. Various plant oils such as rapeseed, castor, soybean, jatropha, and palm oil can be utilized for biofuel production.[16],[17].

Castor beans stand out for their exceptional oil content. Castor oil boasts a remarkable 40-55% oil yield by weight, significantly higher than most common seeds. For comparison, sunflower seeds typically yield 15-20% oil, cabbage seeds 38-46%, and palm kernels 30-60%. Additionally, developing castor beans can be much cheaper [18]. They require roughly half the cost of developing turnips and a mere 25% of the cost for Jatropha. Another key advantage is that castor plants are not suitable for human consumption. This means their use for biofuel production doesn't compete with food crops [11][19]. In fact, castor oil has the potential to fulfill up to 60% of the inedible oil demand for biodiesel. These plants are remarkably resilient, thriving in poor soil conditions and tolerating a wide range of weather conditions [20].

In previous studies, Goi T. Jeong, Don H. Park (2009) stated that the following conditions will generate the maximum yield of castor biodiesel (92 wt.%): a 40-minute reaction at 35.5 °C, with an oilto-methanol molar ratio of 1:8.24, and a catalyst. Concentration of 1.45% KOH by weight of castor oil [21] . Also, D. Lima Silva, et. al (2009) obtained biodiesel from castor oil with a high conversion of 99 wt.% at 30 °C, using 1 wt.% sodium ethoxide, ethanol: castor oil molar ratio of 16:1 for 30 min [22] In 2016, Bhaskar, J., and Soumya, S. accomplished a yield of 91% biodiesel production from castor oil was obtained at 55 °C with a catalyst loading of 14% zinc oxide nano catalyst and a methanol/oil ratio of 12:1 for 50 min [23]. Also, Kira, S. T., Al-Sabbagh, et.al, (2018) achieved yield of 95% caster biodiesel was achieved at 1 wt.% KOH, 60 °C, 9:1 methanol: oil ratio, and a reaction time of 30 min [24].

Many researchers are interested in producing the largest yield of biodiesel, and only a few of them are interested in completing the reaction from converting castor oil by transesterification to obtain pure biodiesel that keeps the engine from corrosion and other problems. In this study, the main objective was

to achieve maximal biodiesel yield with optimal conversion under moderate conditions. A statistical tool was employed to facilitate predicting the results, and a model was devised to align with the research objectives. Utilizing the Design Expert software, a suitable model was constructed to optimize productivity and conversion efficiency employing Response Surface Methodology, focusing on variables such as temperature, reaction time, and methanol concentration in the biodiesel production process. Subsequently, the finest biodiesel specimen was analyzed and benchmarked against global standards.

#### 2.Material And Methods

#### 2.1 Material

The castor seeds utilized in this study were obtained from a private farm in Sharqia Governorate. The oil extraction procedure was carried out in the Mechanical Engineering Department at the National Research Centre in Egypt. Methanol (99.8%), Potassium hydroxide (99%) and The Thin Layer Chromatography (TLC) were acquired from Sigma Aldrich.

#### **Biodiesel Production Process**

Transesterification is a chemical process where vegetable oil, animal fat or seed oils react with an alcohol (like methanol, ethanol, or butanol) as shown in Figure 1. This reaction can happen with or without a catalyst, which can be either mixed throughout the reaction (homogeneous) or separated from the main ingredients (heterogeneous). Among the various homogeneous catalysts, potassium hydroxide, sodium hydroxide, and methoxide are popular choices due to their affordability and effectiveness [16],[25]. The catalyst speeds up the conversion of triglycerides in oils or fats into biodiesel, while also producing glycerin as a byproduct. The type of catalyst used and the reaction conditions can significantly impact the efficiency and selectivity of transesterification [26] [27].

The transesterification process was demonstrated by conducting experimental procedures within a 150 ml glass reactor positioned on a hot plate magnetic stirrer, operating at standard atmospheric pressure. Initially, 100 mL of castor oil was introduced into the reactor and heated to eliminate moisture content. Subsequently, methanol and KOH catalyst were added to the preheated oil and allowed to react until completion. Following the reaction, the mixture was allowed to settle for two hours before undergoing separation using a separating funnel. The resulting biodiesel underwent washing with warm water until the washes appeared clear. To eliminate water and moisture content, the refined biodiesel was dried on a hot plate at 110 °C.

Figure 1: Transesterification reaction of triglycerides.

A comprehensive analysis of various parameters was then systematically conducted to ascertain the optimal reaction conditions, encompassing reaction temperature, duration, and methanol concentration by weight at a 1% KOH catalyst [25],[28] [13]. The levels of the factors were meticulously chosen as shown in Table 1 according to literature review, and initial experimentation. This methodology aided in identifying the optimal process operating conditions.

Table 1: Reaction factors and their levels

Factors	lower limit	Upper limit
Temperature, °C	70	130
Time, min	30	90
Amount of methanol, (%wt.)	10	20

#### Calculation of Synthesized Biodiesel Yield

The mass yield of biodiesel was then determined using Equation (1), following a method described in existing scientific literature [25],[18].

Yield of biodiesel (%)= (Weight of biodiesel (g))/(Total weight of oil in the sample (g)) x 100 (1)

## Calculation of Synthesized Biodiesel Conversion using Thin-Layer Chromatography

Multiple experiments were conducted using castor oil to produce high-quality biodiesel meeting standard specifications. A comprehensive analysis of these experiments, depicted in a figure, identified the optimal conditions for methanol wt.%, temperature of reaction, and reaction time crucial for generating biodiesel of standard quality. The process followed the Thin Layer Chromatography (TLC) method described in Fedosov, Brask, and Xu (2011), involving spotting small sample quantities on specialized TLC plates. These plates were then placed in a solvent container to separate molecules based on polarity and size differences. The biodiesel sample, along with standard samples, were analyzed on silica gel thin layer plates under specific conditions. The plates were developed using a solvent mixture, and the separated components were visualized using iodine vapor [17]. The reaction proceeded until the TLC analysis indicated completion. By comparing the spots of the biodiesel sample to those of the control mixture, we can determine how effectively the reaction converted castor oil to biodiesel. The less similar the spots are, the greater the amount of biodiesel produced. This is how TLC technology helps us monitor and improve the biodiesel production process by allowing us to compare the results of different conversion attempts and choose the most successful one. As a reference, some sources recommend Thin-Layer Chromatography for this purpose [29], [30].

## Modelling with Response Surface Methodology (RSM)

The research utilized the Box-Behnken design, a potent tool in Response Surface Methodology (RSM) incorporated in Design Expert 13 software. This approach facilitated the exploration of how temperature, reaction time, and methanol percentage by weight impact both the quantity of biodiesel derived from castor oil and the efficiency of conversion.

A comprehensive analysis of variance (ANOVA) was performed to evaluate the significance of each factor and their interactions concerning biodiesel production. Furthermore, numerical optimization using Design Expert software was employed to pinpoint the most favorable operational parameters that would result in the highest productivity and conversion efficiency for castor oil biodiesel.

#### **Biodiesel Analysis**

The physical and chemical attributes of the optimized castor biodiesel were assessed, including kinematic viscosity, density, flash point, cloud point, pour point, cetane number, and calorific value. A comparison was made between the properties of the produced biodiesel, standard biodiesel, and diesel fuel to identify notable distinctions[4] [31].

#### 3. Results And Discussion

Optimization with Response Surface Methodology (RSM)

Response Surface Methodology (RSM) guided the analysis and optimization of the biodiesel production process. StatEase's RSM software (version 13.0) was instrumental in this effort. In this study, a Box-Behnken design was chosen to investigate the influence of three critical factors. Three parameters temperature (A), methanol wt.% percentage (B), and reaction time (C) - were varied within specific ranges: temperature from 30 to 90 °C, methanol wt.% from 10 to 20 wt.%, and reaction time from 15 to 60 minutes. Percent biodiesel yield and conversion as the chosen response variables. RSM software planned 27 experiments based on the input. Additionally, Design Expert's numerical optimization function was employed to identify the optimal operating conditions for maximizing biodiesel yield and conversion.

Table (2) suggested 27 experimental runs, where each factor varied within its designated lower and upper

limits. The mass yield of biodiesel (column 5) was calculated using equation (1). Additionally, thin layer

chromatography determined the conversion ratio of Castor oil to biodiesel (column 6).

Table 2: Experimental run designs

Runs	Factor 1 Temperature (°C)	Factor 2 Time (minute)	Factor 3 Conc. Methanol (wt.%)	Response 1 Yield %	Response 2 Conversion %
1	30	15	10	50	40
2	30	15	15	55	55
3	30	15	20	65	60
4	30	30	10	60	50
5	30	30	15	64.34	60
6	30	30	20	69.3	65
7	30	60	10	72.5	65
8	30	60	15	80.5	70
9	30	60	20	85	75
10	60	15	10	77.5	65
11	60	15	15	84.5	75
12	60	15	20	89	75
13	60	30	10	83	80
14	60	30	15	88	80
15	60	30	20	93.5	85
16	60	60	10	80	70
17	60	60	15	84.5	75
18	60	60	20	88.34	80
19	90	15	10	70.5	60
20	90	15	15	74.53	65
21	90	15	20	75	75
22	90	30	10	72.3	55
23	90	30	15	73	60
24	90	30	20	74.3	70
25	90	60	10	75	40
26	90	60	15	76.6	50
27	90	60	20	77	55

#### ANOVA Analysis

Significant factors were identified through extracted probabilities, while ANOVA indicated the quadratic model as optimal for depicting biodiesel production and conversion efficiency. Equations (2)(3) facilitate the effective scaling up of biodiesel production and conversion processes.

Biodiesel Yield % = -29.82704+2.58837 Temperature +0.621471 Time+1.78378 Methanol wt.%-0.007224

Temperature \*Time - 0.015722 Temperature\* Methanol wt.%-0.016470 Temperature² (2)

Biodiesel Conversion % = 46.29630 + 2.68056

 $\label{temperature+1.42857} Temperature+1.27778 Methanol wt.\%-0.013624 Temperature * Time-0.018519 Temperature^2-0.007819 Time^2 (3)$ 

Table 3: ANOVA results for the biodiesel yield response

Source	Sum of Squares	df	Mean Square	F-Value	P-Value	
Model	2579.19	6	429.87	34.84	< 0.0001	significant
A-Temperature	176.32	1	176.32	14.29	0.0012	
B-Time	334.04	1	334.04	27.08	< 0.0001	
C-Methanol wt. %	317.86	1	317.86	25.76	< 0.0001	
AB	295.92	1	295.92	23.99	< 0.0001	
AC	66.74	1	66.74	5.41	0.0307	
$\mathbf{A}^{2}$	1318.29	1	1318.29	106.86	< 0.0001	
Residual	246.74	20	12.34			
Total	2825.93	26				

Table 4: ANOVA Results for the biodiesel conversion response

Source	Sum of Squares	df	Mean Square	F-Value	P-Value	
Model	3531.65	6	588.61	53.91	< 0.0001	significant
A-Temperature	44.00	1	44.00	4.03	0.0584	
B-Time	5.56	1	5.56	0.5089	0.4839	
C-Methanol wt. %	734.72	1	734.72	67.30	< 0.0001	
AB	1052.48	1	1052.48	96.40	< 0.0001	
AC	1666.67	1	1666.67	152.66	< 0.0001	
$A^2$	71.63	1	71.63	6.56	0.0186	
Residual	218.35	20	10.92			
Total	3750.00	26				

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Where A = Temperature (OC), B = reaction time (min), and C = methanol (wt. %). The results of the analysis of variance (ANOVA) in the appropriate statistics table (Table 3, 4) indicate that the model was accurate and that there was a strong and direct relationship between the three factors that were identified for the study and the response. This was supported by the high coefficients of determination (R2) for both the yield and conversion models (0.9127 and 0.9418, respectively). These values indicate a strong fit between the model probabilities and the experimental data, indicating that the probabilities were fairly accurate. The closeness between actual and modelled results is further emphasized by the availability of high adjusted R2 values (0.8865 for production and 0.9243 for conversion efficiency).

#### **Parametric Study**

Figures (2, 3) juxtapose anticipated yields with actual results. The proximity of data points in these figures assures the precision of the model in forecasting yield values. Essentially, the model's predictions closely align with real-world data, indicating a reliable estimation of actual occurrences. The absence of substantial deviations between predicted and observed values enhances our confidence in the model's predictive capacity for future yields. The color gradient in the figures, transitioning from blue to red, signifies the spectrum of biodiesel yield values, with blue denoting lower yields and red reflecting higher yields.

# Influence of temperature and methanol concentration on biodiesel yield and its conversion.

Figure 4 depicts the correlation between temperature, methanol concentration, biodiesel yield, and conversion in a 3-D representation. The x-axis showcases a temperature range spanning from 30 to 90°C, while the z-axis illustrates methanol concentration ranging from 10 to 20 wt.%.

Figure (4, a) illustrates that as methanol concentration and temperature increase, biodiesel vield rises until reaching 70°C. Subsequently, there is a sharp decline in biodiesel yield due to excessive temperatures causing thermal decomposition of the biodiesel or catalyst deactivation, leading to reduced yield. Also, in figure (4, b), it was observed that elevating methanol concentration alongside increasing temperature resulted in a rise in biodiesel conversion until reaching 70°C, after which a sharp decline in biodiesel conversion was noted due to thermal degradation of the biodiesel or catalyst deactivation at higher temperatures, leading to a reduction in the conversion

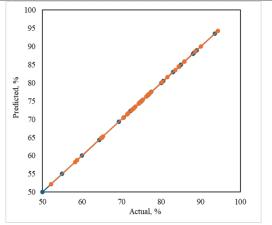


Figure 2: Actual vs predicted values for biodiesel mass yield model.

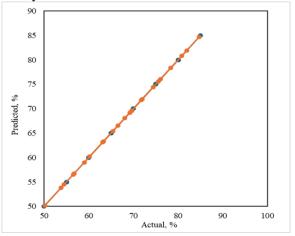


Figure 3: Actual vs predicted values for biodiesel conversion efficiency model.

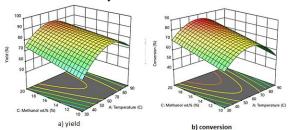


Figure 4: Influence of temperature and methanol concentration on biodiesel yield and its conversion

## Influence of temperature and time of reaction on biodiesel yield and its conversion

Figure 5 depicts the correlation between temperature and time of reaction on biodiesel yield, and conversion in a 3-D representation. The x-axis showcases a temperature range spanning from 30 to 90°C, while the z-axis illustrates time ranging from 15 to 60 wt.%.

In Figure (5, a), it was observed that as the reaction time and temperature increased, there was an initial rise in biodiesel yield followed by a sharp decline. The initial increase is due to enhanced reaction kinetics and improved conversion efficiency.

However, the subsequent sharp decline might result from side reactions occurring at prolonged reaction times and elevated temperatures, leading to decreased biodiesel yield.

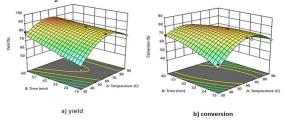


Figure 5: Influence of temperature and time of reaction on biodiesel yield and its conversion

The observed trend in Figure (5, b) suggests that as the reaction temperature increases, there is initially a slight enhancement in biodiesel conversion, likely due to the acceleration of reaction kinetics. However, prolonged exposure to elevated temperatures promotes the prevalence of side reactions and thermal degradation mechanisms, resulting in a subsequent decline in biodiesel conversion efficiency. This phenomenon is attributed to the formation of undesirable by-products and the degradation of reactants, ultimately impairing overall conversion efficiency.

Influence of time of reaction and methanol concentration on biodiesel yield and its conversion Figure 6 depicts the correlation between temperature and time of reaction on biodiesel yield, and conversion in a 3-D representation. The x-axis showcases time of reaction range spanning from 15 to 60°C, while the z-axis illustrates the methanol concentration ranging from 10 to 20 wt.%.

Figure (6, a) demonstrated that increasing time with methanol concentration increases the biodiesel yield. Higher concentrations of methanol enhance transesterification reactions. This increased methanol availability facilitates complete conversion of triglycerides into biodiesel over time, leading to higher biodiesel yields. Prolonged reaction times also promote thorough contact between reactants, further enhancing triglyceride conversion into biodiesel.

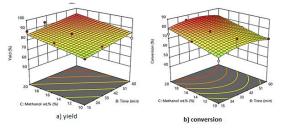


Figure 6: Influence of time of reaction and methanol concentration on biodiesel yield and its conversion

Figure (6, b) demonstrated that increasing time with methanol concentration increases biodiesel conversion then slightly decreases. Initially, higher methanol concentrations accelerate transesterification, increasing triglyceride conversion to biodiesel due to enhanced reactant encounters. However, prolonged reaction times with excess methanol may promote side reactions like soap formation, competing with the main reaction, thus slightly reducing biodiesel conversion.

#### **Optimization Results**

This research aimed to evaluate how three main factors—temperature, methanol concentration (wt.%), and reaction duration—affect the efficiency of biodiesel production, with the primary aim of optimizing both yield and conversion rates. By utilizing Design Expert software, optimization was conducted within predetermined parameter ranges. The software identified the ideal conditions: a temperature of 40.896 °C, a methanol concentration of 20 wt.%, and a reaction duration of 60 minutes. These conditions resulted in significant outcomes, yielding a biodiesel yield of 90.859% and a conversion rate of 82.047%. Moreover, the Desirability score achieved a highly favorable value of 0.896, indicating successful simultaneous achievement of all objectives, as illustrated in Figure 7, where the red dots signify optimal input parameter values, and the blue color represents the optimized output value.

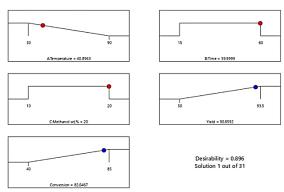


Figure 7: Maximum biodiesel mass yield and conversion efficiency at optimal experimental factors.

## Characterization of the Optimum Biodiesel Produced

Figure 8 shows the Thin-Layer Chromatography (TLC) analysis of the produced castor oil biodiesel. Castor oil's high viscosity makes it unsuitable as a fuel. In contrast, the castor oil biodiesel exhibits lower viscosity, improving its fuel potential. Compared to oil, biodiesel (FAME) is less polar. This allows it to move faster on the silica gel plate when using the hexane, diethyl ether and acetic acid solvent system. Anhydrous iodine crystals were the most

effective way to visualize the separated biodiesel and oil spots on the TLC plate.

The optimal castor oil methyl ester (COME) sample, identified through Design Expert software optimization, underwent characterization for various physicochemical properties following **ASTM** standards. These properties included kinematic viscosity, density, flash point, cloud point, pour point, cetane number, and heating value. The results were then compared with those of fossil diesel and standard biodiesel. This comparative analysis aimed to evaluate the suitability of the produced COME for application in diesel engines and this is shown in table 5.

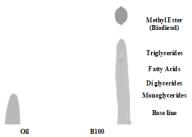


Figure 8: TLC test of optimum Castor oil biodiesel produced.

Table 5: Comparative analysis of physicochemical properties between commercial diesel fuel, Standard ASTM D6751 biodiesel and Castor oil biodiesel.

Property	Commercial Diesel Fuel (D100)	Standard ASTM D6751 Biodiesel	Pure biodiesel prepared from Castor oil (B100)
Density at 15 °C, [kg/m³]	820-860	870 - 890	878
Kinematic viscosity at 40 °C, [mm²/s]	2.0 – 4.5	1.9 - 6.0	15.4
Cloud point, [°C]	(-15) - 5	(-3) – (-12)	-15
Flash point, [°C]	60 - 80	100 - 170	151
Pour point °C	(-35) - (-15)	(-15) – (-16)	-30
Cetane number	40 - 55	48 - 60	43.7
Calorific Value, [MJ/kg]	42-46	-	38.34

#### **Conclusions**

The biodiesel production derived from castor seed oil as an alternate fuel of fossil fuel decreases its harmful impact on environment. Due to its high production quantity that exceeds industrial and agricultural needs, castor seed is considered as a promising source for biodiesel's production. Statistic modeling program was used to obtain the suitable model and study the effect of temperature, time, and methanol concentration on both the biodiesel yield and conversion. TLC technique was used to determine the percentage of the produced biodiesel. The results showed that the transesterification process gives the highest yield (90.859%) and conversion (82.04%) when a sample of oil reacts with 20% wt. methanol while applying at 40oC for 60 mins.

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