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# Experimental optimization of Waste Cooking Oil ethanolysis for biodiesel production using Response Surface Methodology (RSM)

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**Abstract.** Biodiesel production from Waste Cooking Oil (WCO) is the most suitable diesel fuel substitute, due to its cleaner emissions, engine lubricity, nontoxic properties, and renewable sources. This study mainly focused on improving biodiesel experimental production using ethanol and investigating the influence of main operating parameters (ethanol–oil molar ratio, catalyst concentration and stirring speed) on biodiesel yield using Response Surface Methodology (RSM). The problem with using ethanol at the expense of the toxicity of methanol as an alcohol is mainly the separation of glycerol from biodiesel at the end of the transesterification reaction. However, the addition of 5% (v/v) glycerol and 1% (v/v) water at the end of the reaction has been found to aid this separation and improve oil conversion. The optimization of the produced biodiesel is carried out through three factors: Face-Centered-Composite Design (FCCD), building a mathematical model, and statistical analysis, shows that the experimental results agree with the predicted values; they are close to unity with the $R^2$ value (0.9924), indicating the correctness of the model. The optimal conditions of catalyst concentration (1.62 wt%), stirring speed (200 rpm) and molar ratio of ethanol to oil (12.9:1) were obtained, resulting in a biodiesel efficiency of 89.75%. The model was also experimentally validated, achieving about 90% biodiesel yield. The fuel properties of the ethyl ester were investigated and compared successfully with the EN and *ASTM* standards and with baseline local diesel (NA 8110)

**Keywords:** Biodiesel, Ethanolysis, Waste Cooking Oil, Transesterification, RSM optimization.

## 1 Introduction

In recent decades, the depletion of fossil fuel reserves, the global environmental crisis, and the increase in energy demand due to population growth and economic development have attracted great attention worldwide. Many scientific studies have shown that human activity is the main cause of these global problems [1, 2]. Indeed, the massive development of transport, the rapid growth of polluting industries, and excessive energy consumption, have largely contributed to the depletion of natural resources and to environmental degradation through the release of greenhouse gases in particular $CO_2$, responsible for global warming and climate change [1, 3–5]. So, several alternative energy sources derived straightforwardly or in a roundabout from the sun, the wind, hydropower, bioenergy and biofuels or from geothermal energy of the Earth [6] are created and executed to deliver a scope of energy administrations, including power, warming, cooling, and biofuels.

Liquid Biofuels are the most widely used renewable power source in the transport sector [7, 8] where they are blended with conventional fuels such as gasoline and diesel. Most of the worldwide biofuel's productions are bioethanol and biodiesel [9]. Biodiesel is an environmentally friendly nontoxic and biodegradable biofuel [10, 11]. It is being considered as a substitute for petroleum-based diesel fuel in compression ignition (diesel) engines [11–14]. Biodiesel can be obtained using a transesterification process wherein triglycerides from vegetable oils and animal fats react with an alcohol (methanol or ethanol) in the presence of a basic or acid homogenic or heterogenic catalyst [10, 12, 15–18].

Transesterification is the most usually involved technique for the production of biodiesel in the laboratory as well as the industrial scale with a cost-effective and environmentally friendly catalysts [12, 19, 20]. In addition, biodiesel production depends on the cost of raw materials that weighs heavily on its economic viability knowing that price

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of the raw materials accounts for 60–80% of the total production cost of biodiesel [21–23]. An alternative approach to improve the economic feasibility of biodiesel production and contribute to a sustainable society is the use of raw materials that are inappropriate for human consumption. Thus, Waste Cooking Oils (WCO) have been gaining importance as an alternative low-cost feedstock source for biodiesel production [24]. It is economically competitive given its production in huge quantities [25] as byproduct of vegetable oil refineries, food processing and catering industries, trapped grease from treatment plants and animal slaughterhouse [7, 26, 27].

Research studies are progressing in our laboratories, where we are trying to recover WCOs, a very present waste which harms our environment, its recovery remains a necessity. Knowing that; in Algeria, WCOs are considered, according to the *National Waste Agency* (AND[1]), as fatty and non-hazardous wastes. The national legislation (Executive Decree No. 06-104 of February 28, 2006 fixing the nomenclature of waste, including special hazardous waste) classifies them as special waste under the code 20 01. The AND reported 450 519 000 L of WCO generated throughout the nation during 2018, and this huge amount is expected to reach around 550 million liters by 2027. In addition, WCO from both restaurant and household food is recorded 4.12 L per restaurant per day and 0.61 L per inhabitant per month respectively.

Despite the dangers and toxicity of methanol, many studies are based on the use of methanol to produce biodiesel. Methanol is a highly toxic alcohol; its toxicity is caused by its accidental or intentional misuse. This alcohol can lead to serious complications such as metabolic acidosis, visual impairment, and neurological dysfunction. The described brain injury associated with methanol toxicity is hemorrhagic and non-hemorrhagic necrosis of the basal ganglia and subcortical white matter [28]. To address this problem, various works have used ethanol, which has advantages over methanol such as renewable feedstock production and lower toxicity.

The "one factor at a time" classical optimization method is not taking into account interactions between independent variables [29]. To resolve this problem, Response Surface Methodology (RSM) which combines statistical and mathematical techniques to predict response and optimize processes is an effective no frequently used tool [30]. RSM has many advantages in terms of time and cost, it reduces the number of experiments needed to interpret several parameters and their interactions. Face-Centered-Composite Design (FCCD) is one of RSM used to prevent the experiments realized under extreme conditions, it used to study the relationship between variables and optimize their individual and interactional effects [31].

The main purpose of this research is to use the RSM methodology to optimise the operating conditions of biodiesel production from WCO by transesterification process by using low toxic alcohol ethanol. The high ethanol solubility comparing to methanol, complicates the separation of glycerol from the reaction medium. For this reason, glycerol and distilled water at the end of the transesterification reaction was added, in order to facilitate the separation of biodiesel, thus 5% (v/v) of glycerol and 1% (v/v) of water at the end of the reaction. This combination between the several parameters and methods is the key of this study.

The optimal conditions such as catalyst concentration (1.62 wt%), stirring speed (200 rpm) and ethanol–oil molar ratio (12.9:1) were obtained for 89.75% of biodiesel efficiency. The model was also validated experimentally by obtaining a biodiesel yield of about 90%. The study concludes with measurements of physico-chemical properties of the biodiesel produced at the optimum conditions according to the *American Society for Testing and Materials* (*ASTM*) method.

## 2 Materials and methods

### 2.1 Materials

Ethanol, potassium hydroxide and other chemicals were purchased from *Sigma–Aldrich* (99% pure) and the water used was pure distilled deionized. The WCO, originally a mixture of soybean oil (80%) and sunflower oil (20%), was collected free of charge from local restaurants in Algiers and blended to obtain the oil sample. The WCO feedstock was first filtered to remove the impurities then was heated in the oven at 100 °C for 30 min to remove the residual moisture [32]. The characterization of the WCO was then carried out in order to determine its properties that depend on the frying process conditions (duration, temperature, kind of food, etc.). Different parameters were measured such as density, acidity, iodine value, moisture content and Free Fatty Acid rate (FFA). Alkaline catalyzed transesterification process has been used in this study utilizing ethanol and KOH as catalyst. The biofuel resulting from transesterification has been then characterized according to its physicochemical properties by *ASTM* methods including the Kinematic viscosity; Density at 15 °C; Flash Point (FP), Pour Point (PP) and Cloud Point (CP) temperatures; Cetane number and sulfur content.

### 2.2 Statistical design of the experiment

FCCD of RSM is a relevant and widely used methodology reported in the literature for the optimization of biodiesel processes [33].

The Biodiesel production process depends on many variables: mainly reaction time, reaction temperature, stir speed, catalyst concentration, and alcohol–oil ratio. However, to improve biodiesel production yield, it is necessary to develop a better understanding of the interactive effects of these variables on the production process, and identify the optimal parameters values for maximum yield of biodiesel. In this context, FCCD has been used to optimize biodiesel production yield. RSM minimizes the number of experiments that are necessary to find an optimal combination of input variables, reduces research time and overall process costs.

[1] The AND, is an official agency under the authority of the *Ministry of Environment* in charge of the implementation national waste policy.

**Table 1.** Independent factor level settings for a biodiesel production process.

| Factors | Coded symbols | Units | Levels | | |
|---|---|---|---|---|---|
| | | | −1 | 0 | +1 |
| Ethanol–oil molar ratio | $x_1$ | Molar relation | 6 | 12 | 18 |
| KOH concentration | $x_2$ | %w/w | 1 | 2 | 3 |
| Stirring speed | $x_3$ | rpm | 200 | 300 | 400 |

FCCD was applied in this study to explore not only the individual effect of process parameters on biodiesel yield, but also their interactive effects on the response. Three variables, ethanol–oil molar ratio ($\mathbf{x_1}$), catalyst concentration ($\mathbf{x_2}$) and stirring speed ($\mathbf{x_3}$) selected on the basis of related literature research and preliminary experiments in our laboratory [34–38], were considered as independent variables affecting the conversion rate of WCO to biodiesel. The response selected was the yield of ethyl ester ($\mathbf{Y}$%). Table 1 lists the ranges and levels of these independent variables in un-coded and coded values.

The experimental matrix for the factorial design consists of 20 experimental runs including $2^3$ (factorial points), $2 \times 3$ (axial points) and 6 duplicate runs carried out at the center-point level, coded as "0" to estimate the experimental error. All the runs were arranged randomly to reduce systematic error. Experimental data were examined by FCCD utilizing a quadratic polynomial equation to correlate variable response with the independent transesterification parameters. The general form of the equation given below was chosen to fit experimental data:

$$Y = a_0 - a_1 x_1 - a_2 x_2 - a_3 x_3 - a_1^2 x_1^2 - a_2^2 x_2^2 + a_3^2 x_3^2 - a_1 a_2 x_1 x_2 - a_1 a_3 x_1 x_3 + a_2 a_3 x_2 x_3, \quad (1)$$

where, $\mathbf{Y(\%)}$ is the response item of the biodiesel yield, $\mathbf{a_0}$ is the regression coefficient for the intercept, $\mathbf{a_1}$, $\mathbf{a_2}$, $\mathbf{a_3}$ are the first order coefficients of the main effects, $\mathbf{a_{11}}$, $\mathbf{a_{22}}$ and $\mathbf{a_{33}}$ are the coefficients of the quadratic effects, $\mathbf{a_{12}}$, $\mathbf{a_{13}}$, $\mathbf{a_{23}}$, are the coefficients for the interaction between the factors and $\mathbf{x_1}$, $\mathbf{x_2}$, $\mathbf{x_3}$ are the independent variables.

Furthermore, the ANalysis Of VAriance (ANOVA) was carried out to assess goodness-of-fit and the adequacy of the quadratic polynomial regression model. Results were evaluated with various descriptive statistics such as $p$-value and $F$-value. Coefficient of determination $R^2$ and adjusted coefficient of determination $R^2_{\text{adj}}$ were also used. Finally, the RSM analysis was complemented by plotting of response surface to investigate the interactive relationship between the independent variables and predict the maximum biodiesel yield.

### 2.3 Experimental procedure

Initially, the FFA content of the WCO was determined by titration against 0.1 M KOH solution using phenolphthalein as the indicator. In cases where the FFA content is greater than 1%, a pretreatment via an esterification reaction will be necessary in order to reduce the acidity of the WCO, and thus avoid the saponification reaction. The transesterification was performed with 100 g of oil sample in 1 L glass reactor equipped with a magnetic stirrer and adapted to a water bath with a temperature controller. Based on the preliminary studies and the bibliography mainly made in the lab, the reaction temperature and reaction time were kept constant at 60 °C and 60 min, respectively for all the transesterification experiments. Ethanolysis reaction was carried out with different ethanol-to-oil molar ratios and KOH concentration according to factorial design. The solution was mixed until the KOH was completely dissolved in the ethanol. The solution was then added to the oil in the reactor under stirred maintained at desired speed and heated up to reaction temperature (60 °C). The transesterification reaction was conducted until it reached the desired reaction time (60 min). To improve the separation, 5% (v/v) of glycerol and 1% (v/v) of water were added to the mixture under vigorous stirring for 5 min and heating at 60 °C. Based on the preliminary studies in our laboratory, we noted that adding water and glycerol at the end of the reaction permits a rapid separation of the two phases. In contrast, after the water addition no soap was observed. According to Da silva *et al.* [39] this phenomenon was explained by the formation of hydrogen bonds between ethanol and water that are energetically more favorable than the van der Waals interactions between ethanol and biodiesel.

After this step, the total volume of the mixture was then transferred to a separating funnel to allow the complete separation of the two phases based on the difference in density. The upper phase was biodiesel and the lower phase was glycerol. Afterwards, the crude biodiesel was collected and purified by successive washings with distilled water to eliminate minor impurities, and heated above 100 °C in order to remove traces of water, thus improve the quality of the biodiesel (Fig. 1). After each experiment, the reaction yield ($Y$) was calculated per weight of oil giving the equation (2):

$$Y(\%) = \frac{\text{Weight of biodiesel produced}}{\text{Weight of oil used}} \times 100. \quad (2)$$

## 3 Results and discussion

### 3.1 Characterization of WCO

The physical and chemical characterization of the WCO sample was determined and given in Table 2. Values are the mean of triplicate experiments ± Standard Deviation (SD).

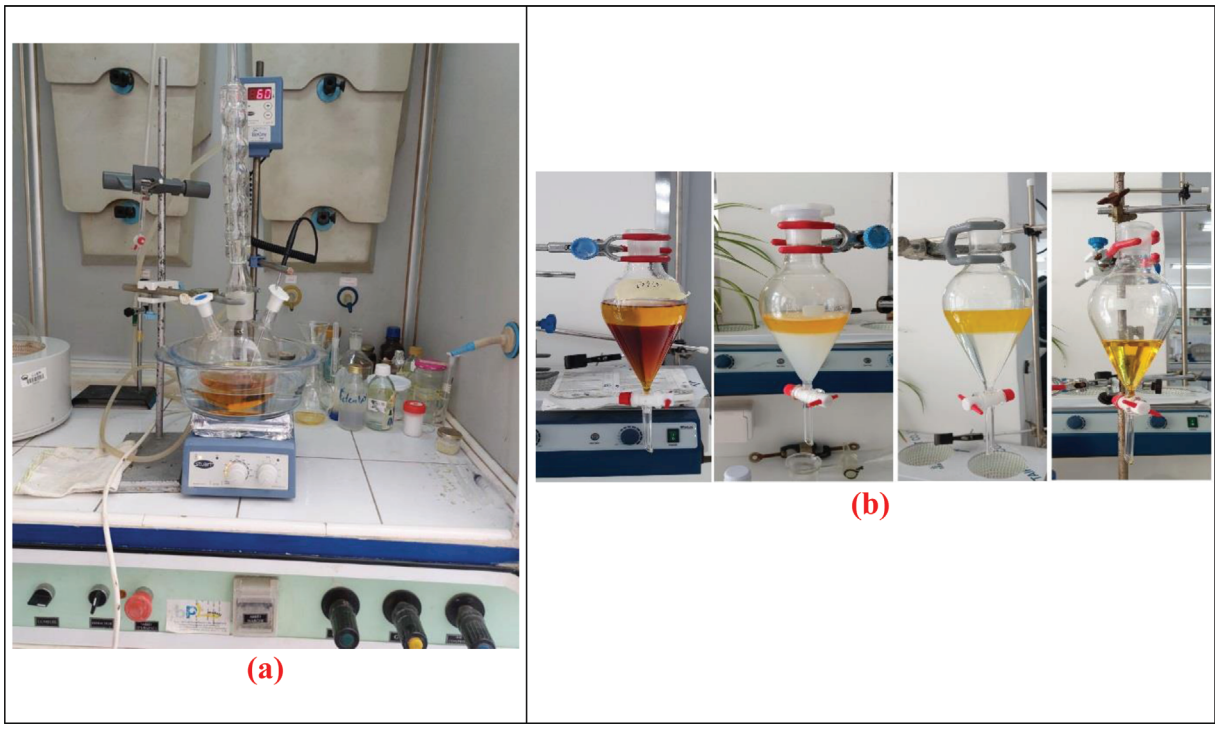


**Fig. 1.** (a) Experimental reactor for biodiesel production. (b) Biodiesel and glycerol separation phases and purification by successive washings.

**Table 2.** Physicochemical properties of WCO.

| Property | Value |
|---|---|
| Water content (vol %) | 0.0252 ± 0.003 |
| Density at 15 °C (g/cm$^3$) | 0.927 ± 0.02 |
| Kinematic viscosity at 40 °C (mm$^2$/s) | 45.5 ± 0.5 |
| Acid value (mgKOH/g) | 1.4 ± 0.06 |
| Free fatty acid (wt%) | 0.7 ± 0.08 |
| Saponification value (mgKOH/g) | 180.6 ± 0.45 |
| Iodine value ((g $I_2$) × (100 g)$^{-1}$) | 103 ± 0.55 |
| HHV (kJ/kg) | 40 480 |

Various studies have reported that the acidity of used cooking oils is a fundamental parameter, as it has tremendous influence on the yield of the transesterification reaction by reducing the efficiency of the process. In alkali-catalyzed reaction method, using potassium hydroxide (KOH) the FFA reacts with the catalyst forming soaps that are highly undesirable and could inhibit the separation of the biodiesel and glycerol during water washing step. Published results [17, 40–42] suggested different levels of FFA recommended for homogeneous base catalyzed transesterification. According to Freedman *et al.* [40], Ma and Hanna [17] and Tiwari *et al.* [42] the FFA content should be less than 1%. However, Dhawane *et al.* [43] indicate that the FFA content should be less than 2% for biodiesel production. Since the FFA value of WCO used as feedstock, in this study it was found to be less than 1 wt%, which implies that WCO could be directly converted into Fatty Acid Ethyl Ester (FAEE) via single-step conversion to biodiesel without undergoing prior treatment. This result was similar to those reported in other studies with WCO [44, 45].

Besides the FFA content, Water content of raw material is also important and considered to be a major concern in the occurrence of the saponification phenomenon which could affect the reaction rate [46]. The water content of the raw material used in this study was 0.025%, lower than that recommended by literature (less than 0.06%) [46].

### 3.1.1 Optimization of transesterification process using RSM

The experimental and predicted results for the model applied to study the effects of three variables, ethanol–oil molar ratio ($\mathbf{x_1}$), catalyst concentration ($\mathbf{x_2}$) and stirring speed ($\mathbf{x_3}$) on biodiesel yield using FCCD, are shown in Table 3.

The obtained experimental data were evaluated by ANOVA and fitted to a second order polynomial equation by multiple regression analysis using JMP Pro 14 software. The resulting FCCD model equation is as follows:

$$Y(\%) = 83.8787 - 0.766996x_1 - 2.555x_2 - 1.495x_3 - 8.53682x_1^2 - 8.59682x_2^2 + 2.50318x_3^2 - 5.19375x_1x_2 - 1.54375x_1x_3 + 3.09375x_2x_3. \quad (3)$$

**Table 3.** Experimental design results for transesterification of waste cooking.

| Run | Ethanol–oil molar ratio ($x_1$) | KOH concentration ($x_2$) | Stirring speed ($x_3$) | Experimental biodiesel yield ($Y_{exp}$) | Predicted biodiesel yield ($Y_{pred}$) |
|---|---|---|---|---|---|
| 1 | 6 | 1 | 200 | 71.00 | 70.42 |
| 2 | 18 | 1 | 200 | 82.05 | 82.36 |
| 3 | 6 | 3 | 200 | 69.20 | 69.51 |
| 4 | 18 | 3 | 200 | 60.10 | 60.67 |
| 5 | 6 | 1 | 400 | 65.00 | 64.33 |
| 6 | 18 | 1 | 400 | 70.50 | 70.09 |
| 7 | 6 | 3 | 400 | 76.20 | 75.79 |
| 8 | 18 | 3 | 400 | 60.30 | 60.78 |
| 9 | 6 | 2 | 300 | 74.77 | 76.10 |
| 10 | 18 | 2 | 300 | 75.55 | 74.57 |
| 11 | 12 | 1 | 300 | 76.00 | 77.83 |
| 12 | 12 | 3 | 300 | 73.70 | 72.72 |
| 13 | 12 | 2 | 200 | 88.50 | 87.87 |
| 14 | 12 | 2 | 400 | 83.90 | 84.88 |
| 15 | 12 | 2 | 300 | 84.00 | 83.87 |
| 16 | 12 | 2 | 300 | 84.03 | 83.87 |
| 17 | 12 | 2 | 300 | 83.98 | 83.87 |
| 18 | 12 | 2 | 300 | 84.00 | 83.87 |
| 19 | 12 | 2 | 300 | 84.01 | 83.87 |
| 20 | 12 | 2 | 300 | 83.98 | 83.87 |

**Table 4.** Estimated regression coefficients for FAEE (%) conversion.

| Source | Coefficient estimate | Standard error | $F$ value | $P$ value |
|---|---|---|---|---|
| $x_1$ | −0.767 | 0.320412 | 5.7303 | 0.0271 |
| $x_2$ | −2.505 | 0.320412 | 61.1221 | <0.0001 |
| $x_3$ | −1.495 | 0.320412 | 21.7703 | 0.0005 |
| $x_1^2$ | −8.445909 | 0.611001 | 191.0770 | <0.0001 |
| $x_2^2$ | −8.755909 | 0.611001 | 205.3610 | <0.0001 |
| $x_3^3$ | 2.5940909 | 0.611001 | 18.0254 | 0.0012 |
| $x_1x_2$ | −5.19375 | 0.358231 | 210.2011 | <0.0001 |
| $x_1x_3$ | −1.54375 | 0.358231 | 18.5706 | 0.0009 |
| $x_2x_3$ | 3.09375 | 0.358231 | 74.5836 | <0.0001 |

Analysis of equation (3) showed that $x_1$, $x_2$, $x_3$, $x_1^2$, $x_2^2$, $x_1x_2$ and $x_1x_3$ have a negative effect on biodiesel yield, which implies that with the increase of the coefficient values the biodiesel yield will decrease. Furthermore, it can be noticed that the concentration catalyst ($x_2$) has a negative effect in the linear term and positive effect in the quadratic term. The results also showed the positive effect on biodiesel yield of the $x_2x_3$ interaction.

ANOVA expressed in terms of $F$-value and $p$-value (Tabs. 4 and 5) was used to assess the statistical significance of independent factors and their interactive effects on biodiesel yield. $p$-value lower then 0.05 was considered significant in surface response analysis [31]. Based on the results of ANOVA the model was statistically significant at confidence level of 95% where a small probability value ($p$-value) and high Fisher value ($F$-value) were observed. $F$-value of 170.816 suggests that model best explains the results, as there is only 0.01% chance that a model $F$-value this large could occur due to noise.

Additionally, it is also important to calculate the values of the coefficient of determination ($R^2$) and the adjusted coefficient ($R^2_{adj}$). The values of $R^2$ and $R^2_{adj}$ were found to be 0.9924 and 0.9856, respectively. These high values of the coefficients indicate that the model is in good agreement with the experimental data. Figure 2 shows the predicted *versus* experimental value of response (biodiesel yield) for

**Table 5.** ANOVA for quadratic model and regression statistics.

| Source | Degrees of freedom | Sum of squares | $F$ value | $P$ value |
|---|---|---|---|---|
| Model | 12 | 1353.2 | 170.816 | <0.0001 |
| Pure error | 5 | 0.0018 | – | – |
| Residual error | 10 | 8.8022 | – | – |

$R^2 = 0.9924$; $R^2_{adj} = 0.9856$; standard error = 0.04.

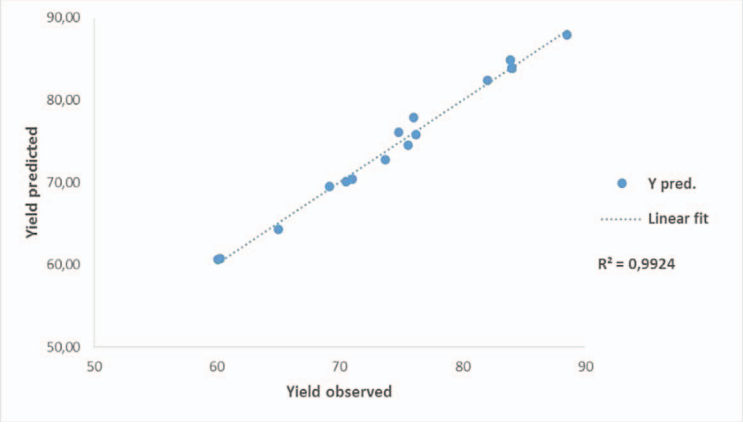


**Fig. 2.** Yield predicted *versus* experimental.

all the runs. The linear regression of fit shows good agreement between the experimental and predicted FAEE yields ($R^2 = 0.99$). The maximum and minimum of FAEE yields achieved were 88.5% and 60.1% respectively.

Moreover, each term in the model was also tested for significance. All terms including the linear, quadratic and interaction terms that affect biodiesel yield are significant since the $p$-value is less than 0.05. From Table 5 it can be seen that among the three variables studied, catalyst concentration provided the major linear effect on biodiesel yield. Its $F$-value is the highest compared to the other factors followed by stirring speed and ethanol–oil molar ratio with a smaller effect. However, the quadratic terms for these two variables (catalyst concentration and stirring speed) have a higher effect than their corresponding linear terms. The results also showed that $x_1x_2$ interaction effect was the most significant. Most studies report that catalyst concentration and ethanol–oil molar ratio have the greatest impact on biodiesel yield [47–50]. However, there are also studies with similar implications that do not have the same conclusions. In each case, the influence of certain factors on the ethanolysis reaction rate and biodiesel yield depends on the type of oil utilized [51]. Based on these results, it can be concluded that the quadratic polynomial model is suitable in the prediction of the yield response with respect to all the variables studied.

### 3.2 Variables effects on biodiesel yield

In order to investigate the effect of different variables on biodiesel yield and their interaction, three-dimensional (3D) response surface plots were generated. The plots were obtained by presenting the response variable *versus* two independent variables while the third was kept constant.

Figure 3a shows the contour plot for the interaction effect of ethanol to oil molar ratio *versus* KOH concentration towards the biodiesel yield. It can be noticed that with the simultaneous increase of ethanol–oil ratio and KOH concentration at stirring speed of 200.01 rpm, the biodiesel yield rises initially and after achieving the maximum value begins to decrease. Encinar *et al.* [47] found in the ethanolysis of used frying oil that increase in the ethanol–oil ratio and KOH concentration increased the yield of alkyl esters, with the best biodiesel yield at ethanol–oil ratio of 12:1 and 1.0% of KOH and beyond these values, the biodiesel yields decrease. High biodiesel yield (88%) was obtained at moderate ethanol–oil ratio and KOH concentration.

Figure 3b depicts the interaction effect between stirring speed and catalyst concentration on biodiesel yield at ethanol–oil molar ratio of 12.9:1. Figure 3c illustrates the interaction effect between stirring speed and ethanol–oil molar ratio on biodiesel yield at catalyst concentration of 1.62 wt%. The results show that at a low level of stirring speed, the yield increased with an increase of the KOH concentration and ethanol–oil molar ratio and then decreased at high levels of the two variables. Nevertheless, the effects of KOH concentration and ethanol–oil molar ratio were less significant at high stirring speed. The High biodiesel yields (>90%) were achieved with low stirring speed at moderated KOH concentration and ethanol–oil molar ratio. In this study, the effect of stirring speed was studied in the range of 200–600 rpm and the optimum for transesterification reaction was found to be 200 rpm. At a higher stirring speed, soap formation was observed. According to

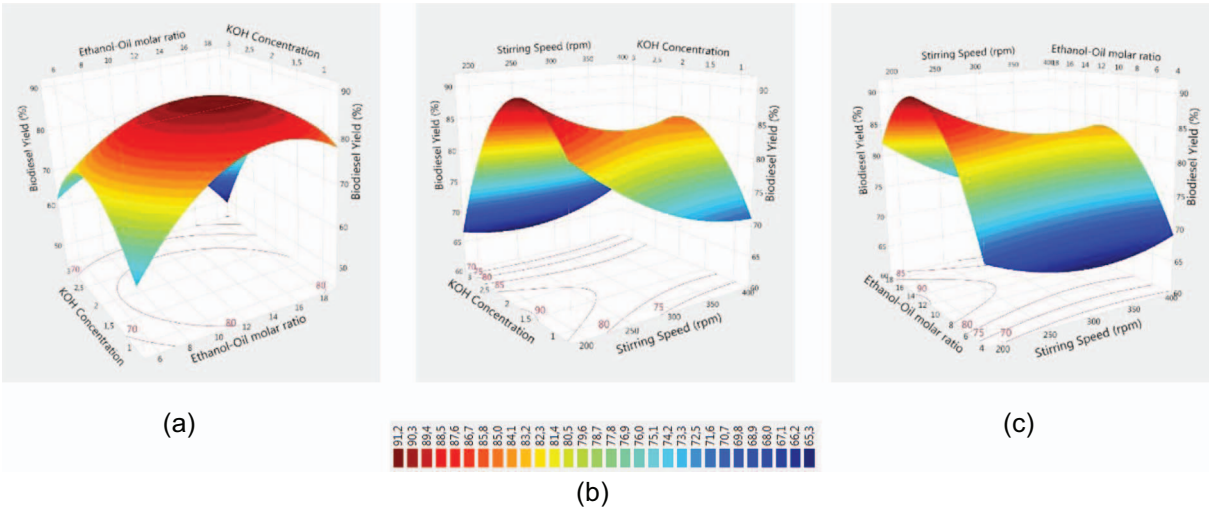


**Fig. 3.** Combined effects of: (a) ethanol–oil molar ratio and KOH concentration, (b) KOH concentration and stirring speed and (c) stirring speed and ethanol–oil molar ratio.

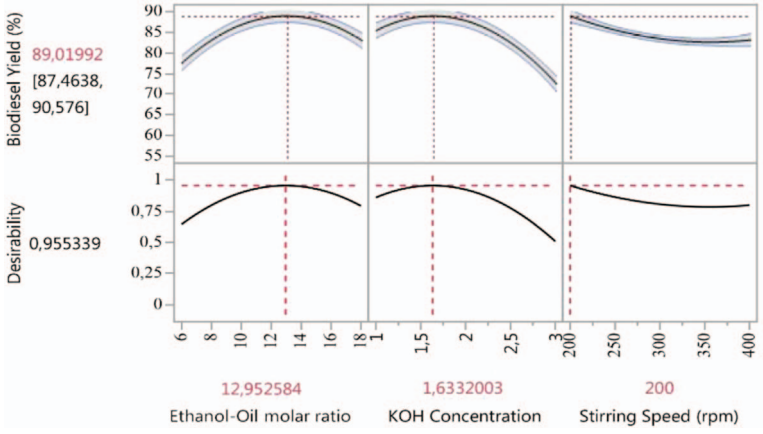


**Fig. 4.** Optimization of multiple responses by composite desirability function.

**Table 6.** Fuel characteristics of FAEE/*ASTM* D6751 – EN 14214 [54] and Diesel NA8110.

| Fuel characteristic | Biodiesel | EN 14214 | *ASTM* D6751 | Diesel NA8110 |
|---|---|---|---|---|
| Density at 15 °C (g/cm$^3$) | 0.884 | 0.86–0.90 | 0.88 | 0.81–0.86 |
| Kinematic viscosity at 40 °C (mm$^2$/s) | 4.2 | 3.5–5.0 | 1.9–6 | – |
| Cetane number (°C) | 47.6 | Min 51 | Min 47 | Min 48 |
| Flash point (°C) | 135 | Min 101 | Min 130 | Min 55 |
| Pour point (°C) | −3 | – | −15 to −10 | −12 to −7 |
| Cloud point (°C) | −3 | – | −3 to −12 | – |
| Distillation temperature °C | 345 | | 360 | Max 350 |
| Acid value (mgKOH/g) | 0.14 | Max 0.5 | Max 0.5 | – |
| Iodine value (mg $I_2$/100 g) | 113 | Max 120 | – | – |
| Water content (%) | 0 | Max 0.05 | – | – |
| Sulphur content (%) | 0.0094 | – | Max 0.05 | Max 0.25 |

Mathiyazhagan and Ganapathi [52] agitation of oil and catalyst mixture enhances the reaction of transesterification. Garlapati *et al.* [53] found in transesterification of *Simarouba glauca* oil that the molar conversion has been found to increase with the increased agitation speed from 100 to 200 rpm with the optimum at 200 rpm.

### 3.3 Optimization of transesterification parameters and model validation

The desirability function was used to optimize the transesterification parameters aimed at obtaining the highest biodiesel yield (Fig. 4). The optimum conditions were found to be ethanol molar ratio, 12.9:1 with KOH concentration of 1.62 wt% at stirring speed of 200 rpm, where the desirability was $D = 0.95$.

To study these conditions experimentally, transesterification of WCO was performed under the reported optimum conditions. The obtained experimental yield, calculated after three replications (standard deviation = 0.25), was an average value of 89.75% compared with the predicted value of 89% from the model. This result shows once again the validity of the correlations and the repeatability of the experimental procedure. It can be concluded, that the findings of the response surface optimization and statistical analyses show a good reliability and practicality of the regression model for making predictions for the industrial production of biodiesel.

### 3.4 Fuel properties of FAEE

The fuel properties of the FAEE obtained under optimized conditions characterized it's physical and fuel properties based on both European Norms EN 14214 and USA *ASTM* D6751 biodiesel quality standards. Additionally, fuel properties of the FAEE obtained in this study were compared with baseline local diesel (NA 8110).

The results listed in Table 6 show that density and viscosity values of the produced FAEE were lower compared to the WCO. The results also show, that all properties of the produced biodiesel sample were in agreement with the biodiesel quality standard limits. According to these results, it can be concluded that WCO Biodiesel can be used as an alternate source of energy instead of diesel.

## 4 Conclusion

In this study, experimental lab scale alkali-catalyzed transesterification using WCO and ethanol was designed to obtain optimal conditions for maximum biodiesel yield.

Even though the use of ethanol led to a separation problem, it was found that the addition of 5% (v/v) of glycerol and 1% (v/v) of water at the end of the reaction facilitates this separation and increases the rate of oil conversion in order to obtain better results and improved efficiency. Mainly FCCD was successfully used in the optimization of the selected operating parameters involved in the biodiesel production process.

An optimum yield about 90% was achieved experimentally with 12.9:1 ethanol–oil molar ratio, 1.62 wt% of KOH concentration at stirring speed of 200 rpm. The biodiesel produced under these conditions conformed to EN and *ASTM* standards. As a conclusion, the biodiesel produced from WCO with bio renewable ethanol is economically and environmentally relevant from the perspective of large-scale biodiesel manufacturing, which opens potential pathways for sustainable biodiesel production in Algeria.

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