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Research Paper

Response surface methodology (RSM) for biodiesel production from waste cooking oil: Study of fatty acid methyl ester (FAME) yield

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The paper aims to improve biodiesel production from used cooking oils using response surface methodology (RSM). The influence of important factors on biodiesel yield has been investigated by employing a 5-level-3-factor central composite design (CCD). The findings revealed that these factors have considerable impacts on the yield. Besides, fixing temperature value had no detrimental influence on the effectiveness of having adequate mathematical model and obtaining good results. According to the analysis of variance (ANOVA), the molar ratio was found to be the most significant factor. Besides, a second-order models (R^2=0.88) were obtained to anticipate the yield as a function of these variables. It is also found that the following ranges of the studied factors can be used to obtain an optimal biodiesel yield: [6.66:1, 8.72:1], [41.62 min, 78.90 min], and [0.91 wt %, 1.79 wt %] for molar ratio, reaction time, and catalyst amount, respectively.

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1. Introduction

Given the accelerated growth in both human population and food consumption, the waste cooking oil (WCO) disposal problem becomes a worldwide concern [1]. Indeed, biofuels made from renewable sources become popular alternative of energy. Depending on the types and the availability of biomasses, the 2^{sd} generation of biofuels is a solution which focuses on using residue biomasses and wastes, including WCO [2]. Indeed, transesterification is widely used process for converting oils to biofuels, it is influenced by various factors that affect the yield [3]. However, many useful statistical methods have been used to optimize the biodiesel yield such as response surface methodology (RSM) [4].

2. Tunisian case study

The yearly consumption of vegetable oils in Tunisia, a North African country, was about 170,000 tons (≈17 kg/person /year). Soybean oil (56%), palm oil (18%), and sunflower oil (10%) make for the majority of this consumption; however, the olive oil, a traditional commodity, accounts only 8%. Indeed, eateries, hotels, university restaurants, hospitals, and refectories are the most common sources of oil waste. The majority is disposed of in landfills (26,700 tons /year), while the remainder is pumped into sewers (35,800 tons / year) [5]. As a result, the current study's general objective was to generate fatty acid methyl esters (FAME) from WCO using potassium hydroxide (KOH). However, the primary goal of the present work was to give a thorough, in-depth analysis of how the new interactions between various factors would affect the production of FAME once the temperature, which has a 47% impact on yield [6], has been fixed. Indeed, it is essential to look at the change in FAME production while keeping the temperature constant in order to understand how the other variables correlate and impact both statistical and modelling research. In this case, a new parameter value range for the first time will be examined for the innovative optimization. Accordingly, the process has been developed and enhanced by using RSM. As a consequence, innovative statistical, graphical, and modelling findings will be presented in this context to enrich the study of WCO biodiesel production.

3. Materials and methods

3.1. Materials

WCO was collected from household wastes. Then, it has been decanted and filtered. Before the experiments, WCO has been heated to the appropriate temperature to eliminate the humidity. Besides, different chemical reagents have been used to carry out these experiments such as

methanol, ethanol (99.97%), hydrochloric acid HCl (37-38%), extra pure KOH, and phenolphthalein indicator (acid/ alkaline).

3.2. Experimental procedure

The experimental setup is shown in Fig.1. After filling the two-neck flask with the necessary amount of WCO, it is placed in the constant temperature bath with all of its supporting equipment and heated to a specified temperature (60°C). The KOH was dissolved in methanol and then heated to (60°C) before being added to the reactor. The reaction started as soon as the KOH-methanol solution was poured and continued until the reaction was completed according to the proper reaction time.

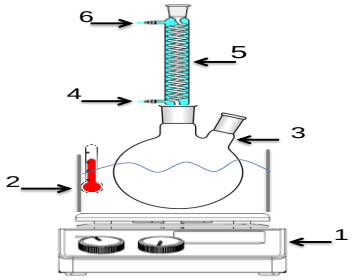

Fig. 1 Experimental equipments (1) agitator- (2) thermal bath-(3) bio-reactor- (4) water inlet- (5) condenser- (6) water outlet

3.3 Separation and purification

After the end of the transesterification reaction, the mixture has been transferred to a separator funnel, where glycerol layer has been separated by gravity overnight. After this layer has been removed, the excess of methanol, KOH, remaining glycerol, and the parasitic by-products like soap have been removed from the methyl ester (biodiesel) layer. In this instance, hot distilled salt water was used to purify the final biodiesel. To prevent hydrolysis reaction, the water volume was halved compared to the obtained biodiesel volume. During the purification process, mixing was avoided to inhibit the hydrolysis reaction which causes biodiesel degradation.

3.4. Calculated Parameters

WCO physic-chemical properties have been analyzed before the experiments. The following have been determined: humidity, H (%), acid value, I_a (mg KOH/1g oil) [7], free fatty acids percentage, $FFAs$ (%) [8], saponification value, I_s (mg KOH/g oil) [9], and average molecular weight, AMW (g/mol) [10]:

$$H(\%)=\left(\frac{m_1-m_2}{m_1-m_0}\right)\text{x}100 \qquad (1)$$

$$I_a = \frac{56.1 x N_1 x V}{P} \quad (2)$$

$$FFAs(\%) = I_a x 0.5 \quad (3)$$

$$I_S = \frac{(V' - V) x N_2 x 56.1}{P} \quad (4)$$

$$AMW = \frac{3x56.1x10^3}{I_s} \quad (5)$$

m_0, m_1, and m_2 are masses (g) respectively of the empty crucible after heating in the oven, the crucible with the test portion before heating in the oven, and the crucible with the test portion after heating in the oven. N_1: is the KOH solution normality (0.1 mol / l), V: is the volume of the poured solution (ml), P: is the mass (g) of the tested sample of oil, N_2: is the HCl normality, V': is the volume of the poured HCl solution (ml) for the blank test, and 56.1 is the molar mass of KOH (g / mol).

3.5. Statistical analysis

The biodiesel yield after the purification steps has been given by:

$$y_{exp} = \frac{V_{FAME}}{V_{WCO}} \quad (6)$$

V_{FAME} is the volume of the pure transesterified oil (in ml), and V_{WCO} is the initial volume of the WCO (i.e. 100 mL).

Indeed, in most RSM cases, the first step is to find a suitable relationship between the variables [11]. If there is a curvature in the system, a second-order equation is commonly used for modelling [12]. This equation is given by [13]:

$$y_{th} = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum \sum_{i<j} \beta_{ij} X_i X_j + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \varepsilon \quad (7)$$

y_{th} is the predicted response, βo, β_i, β_{ij} and β_{ii} are constant coefficients, X_i and X_j are the used factors, and ε is a random error.

The central composite design (CCD) is the most popular second-order designs [14]. It consists of the following points [15]:

- 2^k factorial design whose factors' levels are coded as (-1) and (+1).
- An axial portion consisting of *2k* points arranged so that two points are chosen on the axis of each control variable at a distance of ±α from the design center. We refer to α as the axial parameter. These points are the star points.

- A number, n_0, of replications at the center ($n_0 \geq 1$). This is called the center point portion, which is required for the random error estimations.
- The total number of the design points in a CCD is: $N_t = 2^k + 2k + n_0$.
- The value of α is determined according to the number of factors k: $\alpha = 2^{(k/4)}$

3.6. Studied case

Since the production of biodiesel is affected by many factors, this section presents the range of these factors according to the previous works as described in the following Table 1.

Table 1. Different transesterification conditions

Factor	Conditions	works
Catalyst	NaOH and KOH are the most used between 0.4 and 2% (wt/wt)	[16]
Alcohol	Methanol and ethanol are the most recommended	
	Minimal molar ratio is 3:1	[17]
	Recommended molar ratio is 6:1	[7]
Temperature	Optimal temperature reaction is between 50°C and 60°C	[18]
Mixing	Optimal mixing is 400 rpm	[19] and [20]
Reaction time	Biodiesel reaches its maximum level at a reaction time less than 90 min	[21]
	60 min is more recommended	[22]

The Design Expert.7.0 software was used to evaluate the statistical analysis. The factor number k was equal to 3, α was equal to 1.68, and n_0= 6. Thus, the total number of trials N_t= 20. The coded values of the independent factors are given in Table 2.

Table 2. Experimental ranges and levels of the independent variables

Factors	Range and level				
	Low axial	Low factorial	Center	High factorial	High axial
	-α = -1.68	-1	0	+1	+α = 1.68
X_1	0.96 :1	3 :1	6 :1	9 :1	11.04 :1
X_2	26.4	40	60	80	93.6
X_3	-1.44	0.4	1.2	2	2.54

X_1: molar ratio, X_2: reaction time, X_3 catalyst mass weight.

4. Results and discussions

4.1. WCO physico-chemical characteristics

The physical and chemical characterizations of WCO are reported in Table 3. As it can be seen from this table, the used waste has an acid value equal to 1.122 mg KOH/ g of oil, which is equivalent to 0.561% of FFAs. This percentage is smaller to the obtained value by Hsiao et al. [23], which was equal to 0.74%. The FFAs percentage in palm oil can exceed 23% [19]. This value complies with the requirements for applying base-catalyzed transesterification (*FFA* < 1 mg of KOH/ g of sample) [24]. Moreover, the humidity level was also assessed. It is equal to 0.19%. According to the study of Atadashi et al. [25], the presence of water has a greater negative impact on catalyzed transesterification than the presence of FFAs. Besides, the saponification value is equal to 192.5 mg KOH /g of oil. The obtained value is higher than the refined oil (190.74 mg KOH /g of oil) [26]. Moreover, it is feasible to calculate the WCO average molecular weight by using Equation (5). After the transesterification reaction, the FAME has followed different phases (see Fig.2) to obtain the final product.

Table 3. Waste cooking oil characterizations

Parameter	Value	Unit
Acid value	1.122	(mg KOH /g oil)
Free fatty acids	0.561	(%)
Humidity (%)	0.19	(%)
Saponification value	192.5	(mg KOH /g oil)
Average molecular weight	874	(g /mol)

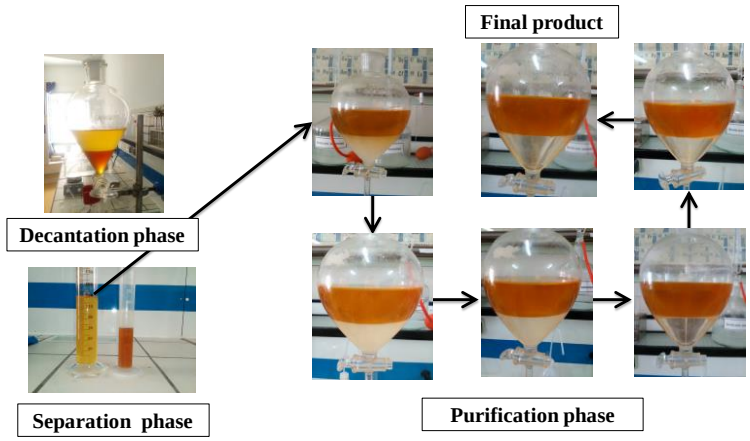

Fig 2. Biodiesel purification phases

4.2. Regression models and statistical analysis

The regression model typically gives the correlation between the dependent and the independent

variables. Series of models have been evaluated as illustrated in Table 4. The linear and the 2FI models exhibit the lowest R^2, adjusted R^2, and predicted R^2 values. Nerveless, the cubic model has the highest R^2 and adjusted R^2 values. Further, this model has the lowest predicted R^2 value (–3.49). However, this model has the ability of creating random equations based on the various inputs. Thus, its equation can't be taken in consideration. The quadratic model, on the other hand, was found suitable, and the obtained regression model created from the experimental data is provided by Equation (10). This model can feet 88% of the experimental values. In fact, Mohammed et al. [27] have found that the R^2 value was equal to 0.80, and they have considered it as acceptable value. Fig. 3 illustrates the predicted yield obtained by Equation (10) versus the experimental yield values. Meanwhile, the relative error between them has been calculated by using Equation (11). The higher relative error has been found when the levels of the factors correspond to: (0, 0, -α) and (-α, 0, 0), for (X_1, X_2, X_3). As a result, relatively low molar ratio, or catalyst mass can induce a reduction in the biodiesel yield. However, the relative error tends towards very low values (i.e. 1.33%), when the factors are adjusted in the central levels (0, 0, 0). To correlate the predicted and the experimental biodiesel yields a correlation between the predicted and the experimental biodiesel yields are obtained. It is given by Equation (12).

$$y_{th-linear} = 0.074 + 0.084X_1 + 0.0003X_2 + 0.094X_3 \quad (8)$$

$$y_{th-2FI} = -0.36 + 0.145X_1 + 0.0004X_2 + 0.281X_3 - 0.0005X_1X_2 - 0.0218X_1X_3 - 0.0009X_2X_3 \quad (9)$$

$$y_{th-quadratic} = -1.166 + 0.35X_1 + 0.005X_2 + 0.97X_3 - 0.0005X_1X_2 - 0.0219X_1X_3 - 0.0009X_2X_3 - 0.017X_1^2 - 2.5\text{x}10^{-6}X_2^2 - 0.29X_3^2 \quad (10)$$

$$Error(\%) = \frac{\left|y_{th} - y_{\exp}\right|}{y_{th}}\text{x}100 \quad (11)$$

$$y_{th-quadratic}(\%) = 0.88y_{\exp}(\%) + 7.87 \quad (12)$$

Table 4. Different tested models

Models	R^2	Adjusted R^2	Predicted R^2
Linear	0.48	0.38	0.167
2FI	0.49	0.26	-0.14
Quadratic	0.88	0.77	0.1
Cubic	0.98	0.93	-3.49

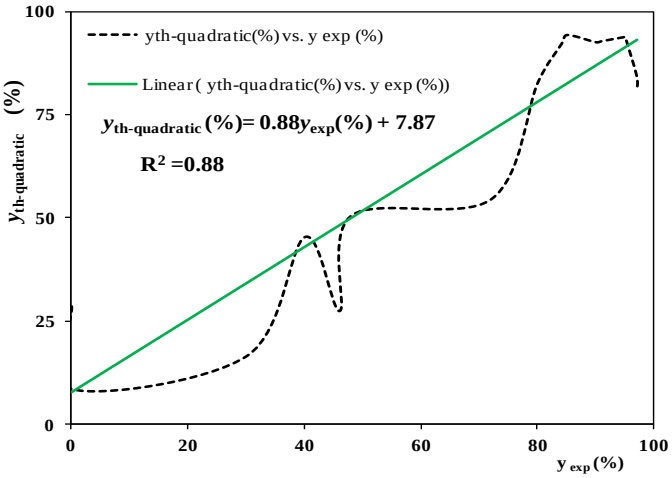

Fig. 3 Theoretical vs. experimental yields

The quadratic model adequacy was tested using the analysis of variance (ANOVA), as shown in Table 5. As it can be seen, the *p*-value is found to be equal to 0.0014 ($p << 0.05$). This indicates that the interactions between the variables are significant. Consequently, the proposed model is correct, and it can be used as a regression tool in this study. The significance of the model indicates that at least one of the independent variables makes a meaningful contribution to the model, as shown in [28]. Besides, the higher model *f*-value is found equal to 8.32, which implies that the model is significant. The interactions X_1^2 and X_3^2 are the most important. The individual factor X_1, on the other hand, has the lowest *p*-value (0.0001), and the highest *f*-value (37.27). As a concluding result, X_1, X_3^2, and X_1^2 are the most significant factors of the model. Many researchers have demonstrated a mixture of significant and insignificant factors, or an interaction between the factors (see for instance: [29]).

Table 5. Analysis of variance (ANOVA) for the response surface quadratic model

Source	Sum of squares	*df*	Mean square	*f*-value	*p*-value prob > *F*	Note
Model	1.75	9	0.19	8.32	0.0014	+*
X_1	0.87	1	0.87	37.27	0.0001	+
X_2	0.0007	1	0.0007	0.03	0.86	-**
X_3	0.07	1	0.077	3.32	0.098	-
$X_1 X_2$	0.0098	1	0.0098	0.41	0.53	-
$X_1 X_3$	0.02	1	0.02	0.94	0.35	-
$X_2 X_3$	0.0018	1	0.0018	0.07	0.78	-
X_1^2	0.33	1	0.33	14.4	0.0035	+
X_2^2	0.00001	1	0.00001	0.0006	0.98	-
X_3^2	0.5	1	0.5	21.01	0.001	+

*(+): significant, ** (-): not significant.

4.3. Interaction between the factors

4.3.1. Interaction between X_1 and X_2

The response surface shown in Fig. 4 represents the interaction effects between X_1 and X_2 by keeping the other variables at their central level. According to this study, the highest biodiesel yield has been simultaneously obtained when X_1 was between 7.5:1 and 9:1, and X_2 was between 40 and 50 min. Thus, increasing X_1 has a positive effect on biodiesel production when X_2 tends towards lower values (i.e. 40 min). This response surface shows that the advantage of using higher X_1, significantly induces X_2 reduction. Similar results have been obtained by Winoto et al. [30]. This is due to the stoichiometry of the transesterification, which includes the conversion of one ester and alcohol into another ester and another alcohol, with an excess of alcohol being utilized to speed up the process. As a result of the higher alcohol content, more biodiesel is converted in less time [31].

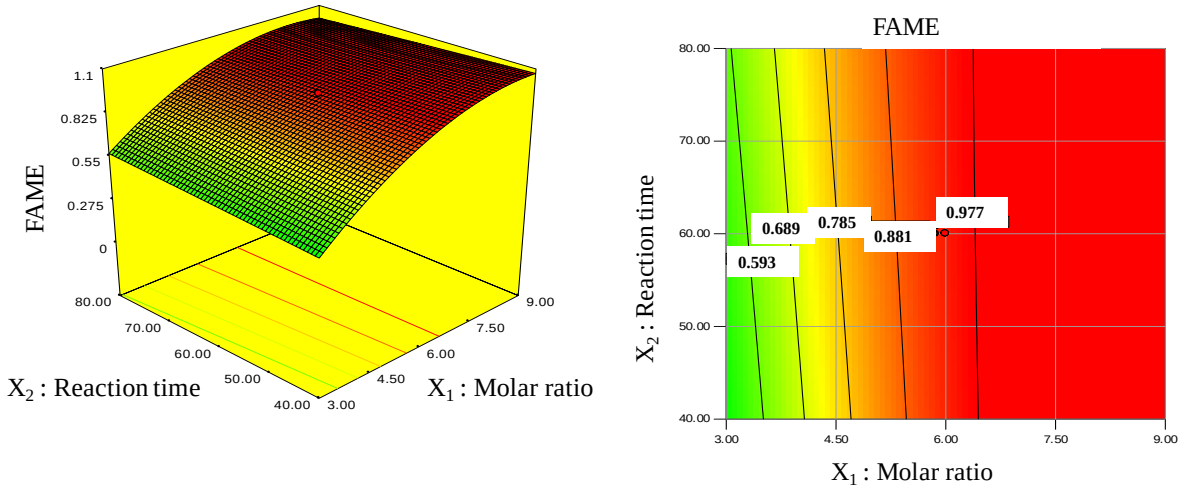

Fig. 4 Interaction between molar ratio and reaction time

4.3.2. Interaction between X_1 and X_3

Fig. 5 shows the FAME yield as a function of X_1 and X_3. Higher yields can be observed when X_1 becomes greater than 6:1, and the X_3 is more than 0.8% (wt/wt). For lower X_1 values (<6:1), the yield is improved when X_3 increases. However, a greater yield was consistently obtained when X_1 was adjusted at a higher level. In this case X_1 was between 7.5:1 and 9:1. On the other hand, the simultaneous decrease of both X_1 and X_3 induces lower FAME yields, and in the contacted surfaces between oil and alcohol [32]. In fact, due to the mass transfer resistance, a high catalyst concentration leads the reactant mixture to become more viscous, resulting in a drop in the reaction rate [33] and soap production [34].

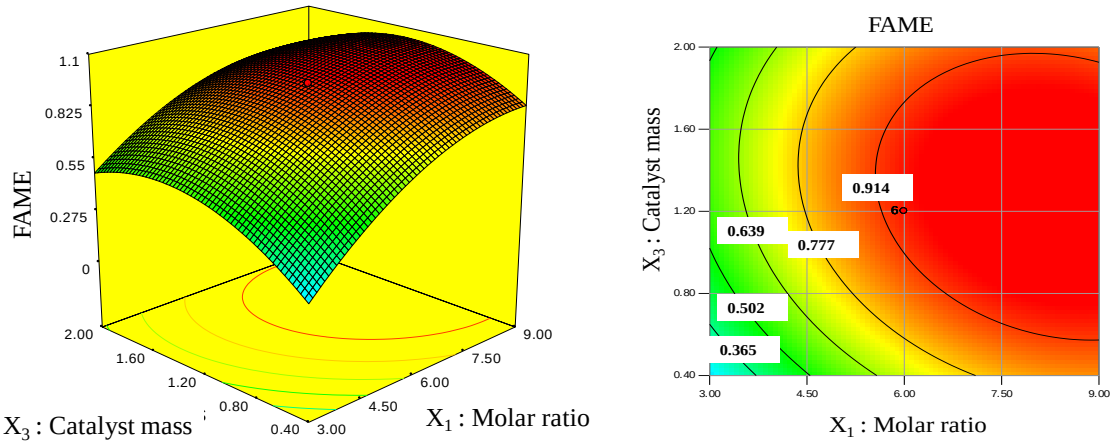

Fig. 5 Interaction between molar ratio and catalyst mass

4.3.3. Interaction between X_2 and X_3

Interaction between X_2 and X_3 on the yield was evaluated. It is found that there is no significant interaction between them (i.e. *p*-value = 0.7873). As illustrated by the 2D plot of Fig.6, for a fixed X_3, variation of X_2 has no significant effects on the yields. This negligible effect can be observed when X_2 is ranging between 60 and 80 min. The variation of the KOH mass has an impact. In this case, fixing X_2 and increasing KOH mass at a certain limited value has a positive effect on the response. In fact, for KOH mass between 0.4 and 1 wt %, the yield will increase from 71% to 90%. Indeed, transesterification cannot occur without the presence of an adjusted catalyst mass [35]. Besides, for X_3 ranging between 1 and 1.8 (wt %), the yield shows practically a constant value at X_2 value. This range can be considered as an optimal X_3 mass catalyst to produce FAME. Accordingly, producing higher biodiesel can be achieved at shorter reaction times. According to the 2D plot in Fig. 6, it is clear that after this catalyst amount, the biodiesel yield decreases to less than 85%. Indeed, a larger X_3 promotes soap production and higher viscosity in reaction mixture, causing a reduction in FAME yield [36]. In fact, the inclusion of catalyst can impact the reaction medium even by adjusting the temperature and mixing speed. In reality, vaporization of the used alcohol is caused by the temperature. As a result, introducing catalyst increases the viscosity of the mixture, lowering the velocity, and amount of reactants in the reaction.

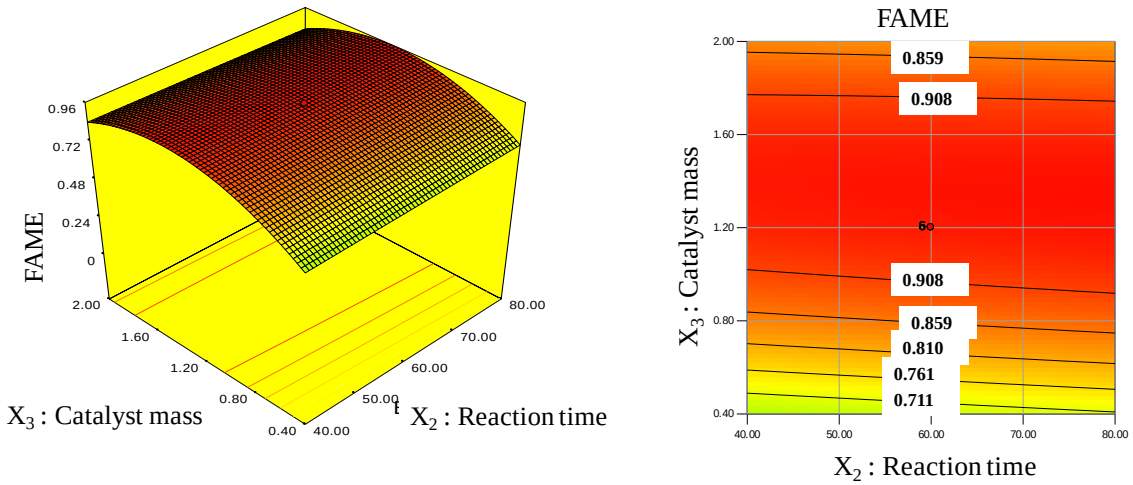

Fig. 6 Interaction between reaction time and catalyst mass

5. Optimization of the operating factors

The optimal yield may vary depending on the factors ranges. Accordingly, the selection of the factor's intervals is crucial for constructing the best yield production. These factors have been chosen according to their important effects on biodiesel production. However, reaction temperature and agitation speed have been fixed. According to the experimental conditions, the intervals of the studied three factors were as follows: $X_1 \in$ [3:1, 9:1], $X_2 \in$ [40 min, 80min] and $X_3 \in$ [0.4 wt%, 2 wt %]. After running the software, various options to produce nearly 100 % of the biodiesel were obtained. Fig. 7 depicts one of the previously described solutions. According to this predicted yield, the experimental yield can be determined using Equation (12). Indeed, the obtained optimal ranges of the factors are as follows: [6.66:1, 8.72:1], [41.62 min, 78.90 min] and [0.91 wt %, 1.79 wt %] for X_1, X_2, and X_3, respectively.

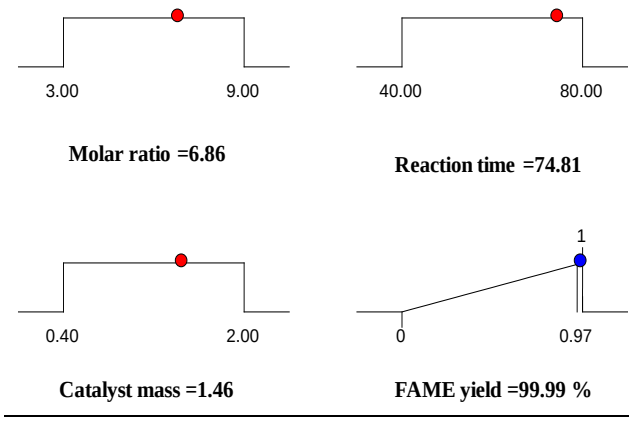

Fig. 7 Optimized factors for higher biodiesel yields

6. Conclusion

Optimizing the operating factors and investigating the kinetics of the transesterification reaction for the generation of biodiesel by using WCO were the main aims of the present study. The followings are some points that may conclude this research:

- The synthesis of FAME from WCO using KOH showed an improvement by using CCD.
- A quadratic equation (R^2=0.88) was developed for biodiesel prediction yield.
- According to this study, fixing temperature has no negative impact on the effectiveness to have good mathematical simulation outcomes.
- The molar ratio has the most significant influence on biodiesel output.
- Different optimal ranges have been obtained by the optimization.
- This research has demonstrated that some research-based optimum values are well validated by this CCD for production, optimization, and simulation.
- As limitation of this work, it is recommended to valid experimentally the theoretical optimized yields.

Declarations

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Availability of data and materials: The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Conflict of Interest: The authors declare no conflict of interest.

Competing Interests: The authors declare no competing interests.

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