

Parameter Optimization for the Biodiesel of *Xanthoceras sorbifolium* Oil and Determination of Fatty Acid Methyl Ester

Liwen Pang,^a Shanshan Lv,^b Kaiquan Li,^b Lijun Ding,^{b,*} and Yinan Hao^c

Biodiesel was prepared from *Xanthoceras sorbifolium* Bunge oil and methanol by transesterification with NaOH as the catalyst. The central composite design (CCD) was utilized to optimize its process, and Design Expert software was applied to perform a fitting regression. The best process conditions were determined as: NaOH 0.9%, molar ratio of methanol and oil 7:1, and reaction temperature 62 °C. Under these conditions, the yield of biodiesel was 89.0%. The composition of *Xanthoceras sorbifolium* Bunge oil-based biodiesel was ascertained by qualitative and quantitative analysis using gas chromatography.

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Keywords: *Xanthoceras sorbifolium* Bunge oil; Biodiesel; Fatty acid methyl ester; Optimization

Contact information: a: Inner Mongolia Vocational College of Chemical Engineering, Hohhot 010070, China; b: College of Science, Inner Mongolia Agricultural University, Hohhot 010018, China; c: College of Materials Science and Art Design, Inner Mongolia Agricultural University, Hohhot 010018, China;

* Corresponding author: dlj78@163.com

INTRODUCTION

The depletion of fossil fuels and the increase in greenhouse gas emissions have a substantial impact on environmental sustainability, which can be reduced through the use of renewable energy (Booramurthy *et al.* 2019). Biodiesel is one of the best alternative fuels for petroleum (Munir *et al.* 2019). It is environmentally friendly, renewable, and harmless (Hao *et al.* 2021). Many kinds of non-edible oils, such as wild chlorella (Mao *et al.* 2019), wild mustard canola oil (Al-Dobouni *et al.* 2016), and bitter almond oil (Fadhil and Mohammed 2018), have been successfully synthesized from different raw materials for producing biodiesel. Furthermore, *Xanthoceras sorbifolium* seed oil can also be synthesized for biodiesel (Ding *et al.* 2013). *Xanthoceras sorbifolium* Bunge is a genus of plants, deciduous trees, or shrubs, and originally produced in northern China; it is unique among China's rare woody oil plants (Wang *et al.* 2020). The seed content was determined as 30.4%, and the kernel oil content was 66.4% after peeling, and the unsaturated fat content was 94% (Zhao *et al.* 2020). It basically contains no nitrogen (Xu *et al.* 2022).

The preparation methods of biodiesel include direct mixing, microemulsion, high temperature cracking, and transesterification, where transesterification is more effective, simple, and feasible (Rong *et al.* 2021). When the content of fatty acid methyl ester in biodiesel is less than 95%, there may be an adverse effect on the combustion and storage performance of biodiesel (Chen *et al.* 2013). Therefore, the content of fatty acid methyl ester is one of the important indexes to evaluate the quality of biodiesel.

At present, there are few research reports on the preparation of biodiesel from *Xanthoceras sorbifolium* seed oil. In this paper, biodiesel was prepared by transesterification of *X. sorbifolium* seed oil and methanol using NaOH as catalyst, and the content of fatty acid methyl ester was determined by gas chromatography. This work provides a reference for the development and application of *X. sorbifolium* seed oil.

EXPERIMENTAL

Materials

For the manufacturing of biodiesel, the *Xanthoceras sorbifolium* Bunge seed was employed as the primary ingredient. *Xanthoceras sorbifolium* was purchased from Chifeng City, Inner Mongolia Autonomous Region, China. Petroleum ether (60 to 90 °C) was purchased from Fuyu Company in Tianjin, China. Methanol and sodium hydroxide were purchased from Wind Ship Company in Tianjin, China. One of these, sodium hydroxide, serves as the reaction's catalyst. The anhydrous sodium sulfate was purchased from Yaohua Company, Tianjin, China. All above-mentioned reagents were of analytical grade.

The authors purchased fatty acid methyl ester standards from Bailingway Company in Beijing, China, primarily including methyl palmitic acid, methyl stearate, methyl oleate, methyl linoleate, methyl linolenic acid, and methyl erucate.

In the experiment, the authors used a DF-101S magnetic stirrer made by Gongyi Yuhua Company, Henan province, China; a China Tianjin Kangda Company KDF-2311 crusher; RE-52A rotary evaporation instrument from Shanghai Yarong Company, China; Quintix 224 analytical balance from Sartorius, Beijing, China; China Ningbo Haishu Co., Ltd. KS-300EI ultrasonic cleaning instrument; China Shanghai Yiheng Company DZF-6210 vacuum drying oven; and a SHZ-III circulating water vacuum pump made by Dufu Company in Zhengzhou, Henan Province, China. In addition, the authors used a gas chromatograph from Shimadzu Company, Tokyo, Japan.

Methods

Preparation and yield calculation of biodiesel

The seeds were shelled to obtain the kernel. After crushing and screening, the kernel oil was extracted with petroleum ether (60 to 90 °C). After separation, the kernel oil was pre-treated with impurity removal, acid removal, and degumming and used to prepare biodiesel.

The seed oil of *Xanthoceras sorbifolium* was placed in a reaction vessel. NaOH was dissolved in methanol, then added into the reactor, and then the transesterification was performed by magnetic stirring and heating in a constant temperature water bath. At the end of the reaction, the unreacted methanol was removed and placed in the separating funnel. Moreover, the separation, recycling, and refining of glycerol was completed through repeated washing and refining. Ultimately, transparent pale-yellow biodiesel was obtained by drying with anhydrous Na₂SO₄.

Because the molecular weight of *X. sorbifolium* oil is large, the difference between the molecular weight of *X. sorbifolium* oil and 3 mol fatty acid methyl ester oil is negligible. Therefore, the theoretical mass of biodiesel is equivalent to the initial mass of citrus oil (Qiao 2008). The yield was calculated as follows:

$$\text{Biodiesel yield } Y (\%) = \frac{\text{Biodiesel quality}}{\text{Quality of } Xanthoceras \text{ sorbifolium} \text{ Bunge seed oil}} \times 100\% \quad (1)$$

Preparation of *Xanthoceras sorbifolium* Oil-based Biodiesel

Single factor pretest

The effects of four factors on the yield of biodiesel were investigated by a single factor pretest. The experimental factors and levels are shown in Table 1.

Table 1. Factors and Levels of Single Factor Experiment

Experimental Factor	Molar Ratio of Alcohol to Oil	Extraction Temperature (°C)	Catalyst Dosage (g/100 g)	Reaction Time (min)
Test Level	4:1	50	0.6	40
	5:1	55	0.8	60
	6:1	60	1.0	80
	7:1	65	1.2	120
	8:1	70	1.4	140

Central composite experimental design

According to the single factor pretest results, the central composite design (CCD) method was utilized to optimize the conditions of preparation. The molar ratio of methanol to oil, reaction temperature, and catalyst dosage were expressed by X_1 , X_2 , X_3 , and by -1, 0, 1, which represents the low, middle, and high levels of each variable, respectively. The independent variable was coded according to the equation $x_i = (X_i - X_0) / \Delta X$, and the response value was biodiesel yield Y (%). Test factors and levels are shown in Table 2.

Table 2. Codes and Levels of Experimental Factors on Central Composite Design

Test Factor	x_i Factor Level Coding				
	-r	-1	0	1	+r
Molar ratio of alcohol to oil (X_1)	4.3:1	5:1	6:1	7:1	7.7:1
Extraction temperature (X_2) (°C)	51.5	55	60	65	68.4
Catalyst dosage (X_3)/(g/100g)	0.66	0.8	1.0	1.2	1.34

Note: The r value was 1.682 when $p = 3$ and $m_0 = 6$

Determination of Fatty Acid Methyl Ester in Biodiesel

Gas chromatography conditions and qualitative analysis of biodiesel components

A JW—DBwax capillary chromatography column (30 m × 0.25 mm × 0.25 μm) was used with a sample size of 1 μL. The temperature of the column oven was 250 °C. The carrier gas was high purity nitrogen, with stigma pressure of 60 kPa. The column incubator was heated according to a programmed method in which the initial temperature was 170 °C, holding for 0.5 min, at a heating rate of 5 °C/min to 220 °C, then a holding time of 5 min. The hydrogen flow rate was 11.2 mL/min, air flow rate was 320 mL/min, and the detector was FID, with a temperature of 250 °C.

Figures 1 and 2 show the chromatograms of the mixed fatty acid methyl ester and *Xanthoceras sorbifolium* oil-based biodiesel. Under the conditions described in the

previous paragraph, the chromatography peaks 1, 2, 3, 4, 5, and 6 are methyl palmitate, methyl stearate, methyl oleate, methyl linoleate, methyl linolenic acid, and methyl erucate, respectively.

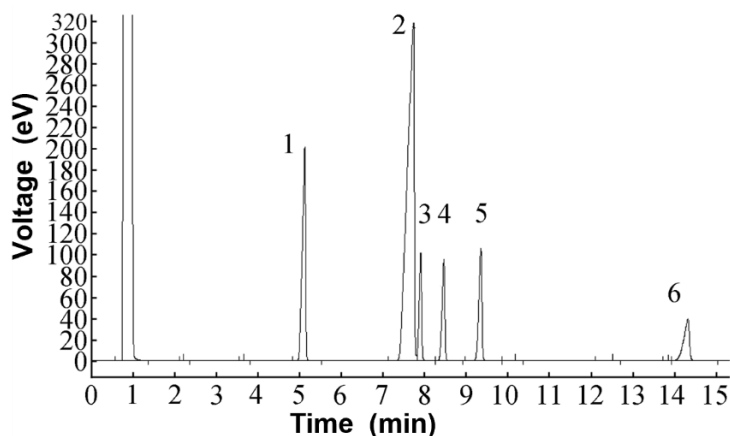


Fig. 1. GC chromatogram of standard solutions

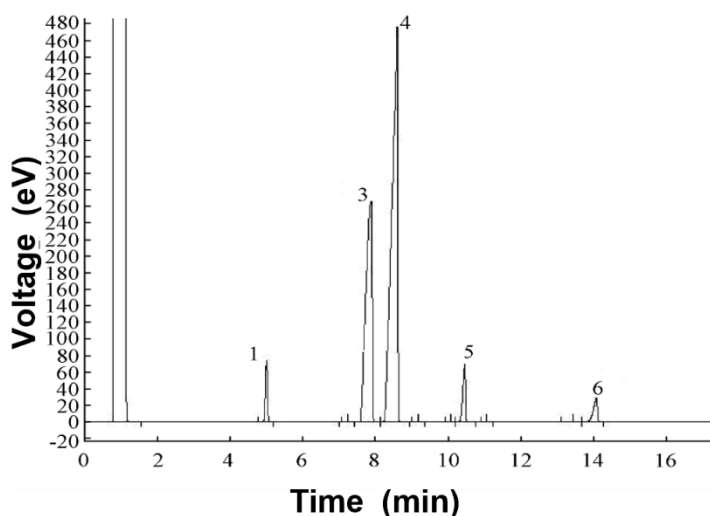


Fig. 2. GC chromatogram of biodiesel made by *Xanthoceras sorbifolium* seed oil

The nature and quantitative analysis of fatty acid methyl ester

Qualitative analysis: The solutions of methyl palmitate, methyl stearate, methyl oleate, methyl linoleate, methyl linolenic acid ester, and methyl erucate hexane were analyzed under the above chromatography conditions. The mixture of the above six fatty acid methyl esters was prepared in a certain proportion and analyzed under the same chromatography conditions as the biodiesel samples, and the retention time was compared, then the fatty acid methyl ester composition of the biodiesel samples was determined (Fang and Zeng 2005).

Quantitative Analysis: The internal standard method was used for analysis. The internal standard was methyl stearate, and the standard curve was established for quantitative calculation (Liu *et al.* 2006).

RESULTS AND DISCUSSION

Preparation and Optimization of *Xanthoceras sorbifolium* oil-based Biodiesel

Analysis of single factor pretest results

The effects of alcohol-oil molar ratio, extraction temperature, catalyst dosage, and reaction time on the yield of biodiesel were investigated by a single factor pretest. The results are shown in Fig. 3.

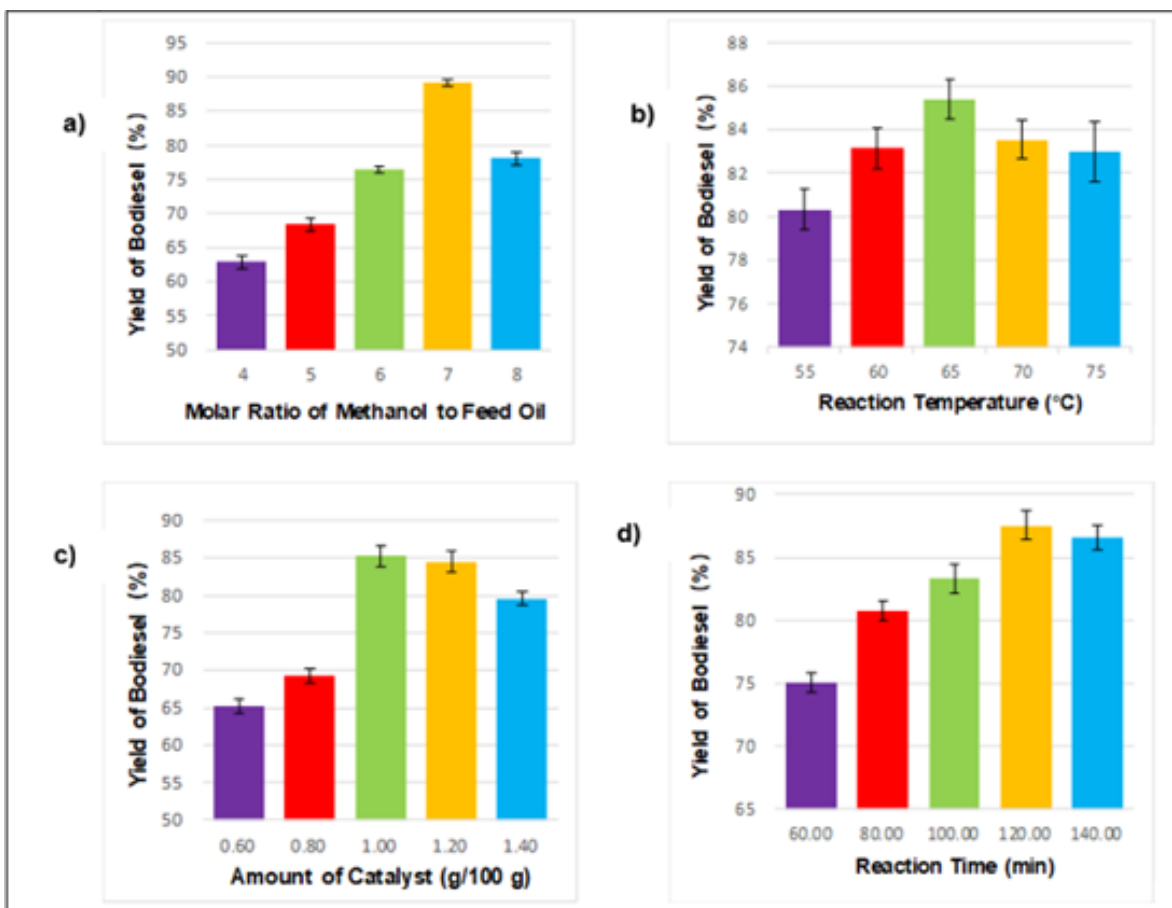


Fig. 3. a): Effect of alcohol to oil molar ratio on biodiesel yield; b): Effect of reaction temperature on biodiesel yield; c): Effect of catalyst amount on biodiesel yield; d): Effect of reaction time on biodiesel yield

The results of the single factor experiment show that the optimum reaction temperature was near 65 °C, which is close to the boiling point of methanol. The temperature was low and the optimum molar ratio of methanol to oil should be 7:1; hence, the increase of molar ratio of methanol to oil can lead to an effective transesterification progress. When methanol and feed oil have an excessively high molar ratio, the glycerol is dissolved in methanol, and it is difficult to separate from the reaction system (Fabbri *et al.* 2007). The optimal catalyst dosage was near 1.0%. The increase of catalyst dosage promotes the saponification reaction, whereby the viscosity of the reaction system is increased. This tends to make it difficult to separate glycerol, and the biodiesel yield is decreased. The optimum reaction time was 120 min. When the reaction time was more than

120 min, the saponification reaction was intensified, the separation and refining were difficult, and the yield of biodiesel was decreased.

Central composite test results analysis

Table 3 shows that optimum design of the test and the test results.

Table 3. Experimental Design and Results of Central Composite Design

Test Serial Number	Molar Ratio of Alcohol to Oil (X1)	Extraction Temperature (X2, °C)	Catalyst Dosage (X3, g/100 g)	Biodiesel Yield (Y, %)
1	-1(5:1)	-1(55)	-1(0.8)	68.69
2	1(7:1)	-1	-1	79.61
3	-1	1(65)	-1	74.49
4	1	1	-1	89.23
5	-1	-1	1(1.2)	77.72
6	1	-1	1	78.63
7	-1	1	1	82.54
8	1	1	1	83.59
9	-1.682(4.3:1)	0(60)	0(1.0)	79.91
10	1.682(7.7:1)	0	0	87.09
11	0(6:1)	-1.682(51.5)	0	77.19
12	0	1.682(68.4)	0	80.08
13	0	0	-1.682(0.66)	80.93
14	0	0	1.682(1.34)	82.94
15	0	0	0	89.31
16	0	0	0	85.67
17	0	0	0	86.19
18	0	0	0	85.19
19	0	0	0	88.98
20	0	0	0	84.35

Design Expert is software provided by Stat-Ease Inc. (Minneapolis, MN, USA) for performing experimental designs and is the most widely used software in the published papers on response surface (RSM) optimization trials. The software version used in this experiment is Design Expert 11. Using Design Expert software, the experimental results in Table 3 were regressed, and the quadratic polynomial regression equation of biodiesel yield to the molar ratio of methanol to oil, reaction temperature, and NaOH dosage was obtained as Eq. 2:

$$Y = -619.70498 + 29.36364X_1 + 15.84121 X_2 + 237.17658 X_3 + 0.099000 X_1 X_2 - 14.81250 X_1 X_3 - 0.70500 X_2 X_3 - 1.46538 X_{12} - 0.12742 X_{22} - 50.46719 X_{32} \quad (2)$$

The significance test and variance analysis of the coefficients of the model are separately shown in Tables 4 and 5.

Table 4 shows that the first terms X_1 and X_2 of the regression equation had a noticeable effect on the yield of biodiesel, while X_3 had no appreciable impact. From Table 5, it can be seen that $F_{\text{misfit}} = 1.71 < F_{0.05}(5,5) = 5.05$, which indicates that the difference of misfit of the model equation was not significant, and that the model was appropriate $F_{0.01}(9,10) = 4.94 < F_{\text{equation}} = 9.24$, which shows that the regression equation model was valid.

Table 4. Significance Test Results of Regression Equation Coefficients

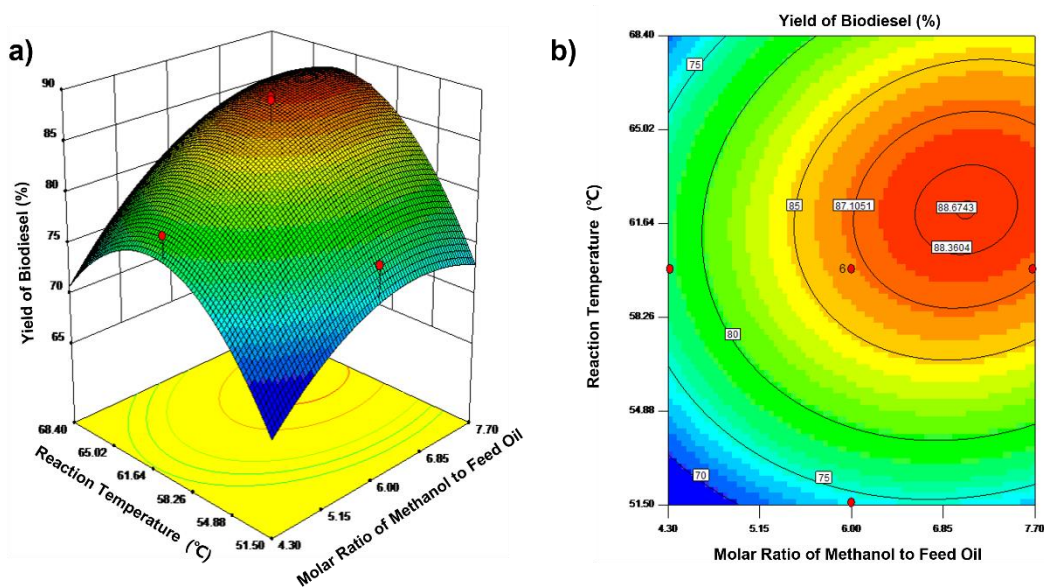
Coefficient Term	Sum of Squares	Degree of Freedom	Mean Square	F-value	P-value	Significance
X1	115.38	1	115.38	20.21	0.0012	**
X2	66.17	1	66.17	11.59	0.0067	**
X3	14.03	1	14.03	2.46	0.1481	
X1 X2	1.96	1	1.96	0.34	0.5709	
X1 X3	70.21	1	70.21	12.30	0.0057	**
X2 X3	3.98	1	3.98	0.70	0.4235	
X12	30.95	1	30.95	5.42	0.0422	*
X22	146.23	1	146.23	25.61	0.0005	**
X32	58.73	1	58.73	10.28	0.0094	**

Table 5. Variance Analysis of Regression Equation

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-value	Pr > F	Significance
Regress	474.67	9	52.74	9.24	0.0009	**
Residual error	57.10	10	5.71			
Misfit	36.01	5	7.20	1.71	0.9335	
Error	21.09	5	4.22			
Summation	531.77	19				

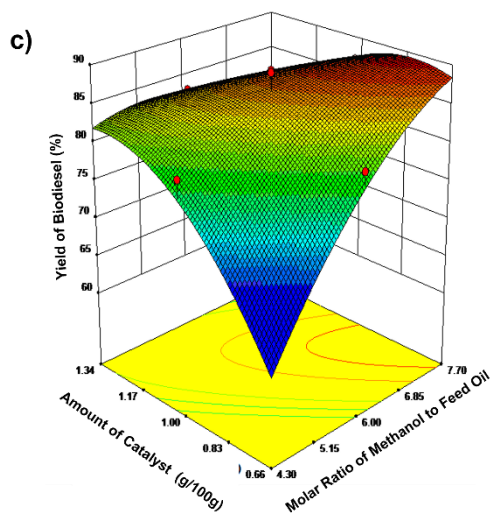
Interaction analysis of each factor

The response surface and contour of the influence of the factors and their interactions on biodiesel yield are shown in Fig. 4.

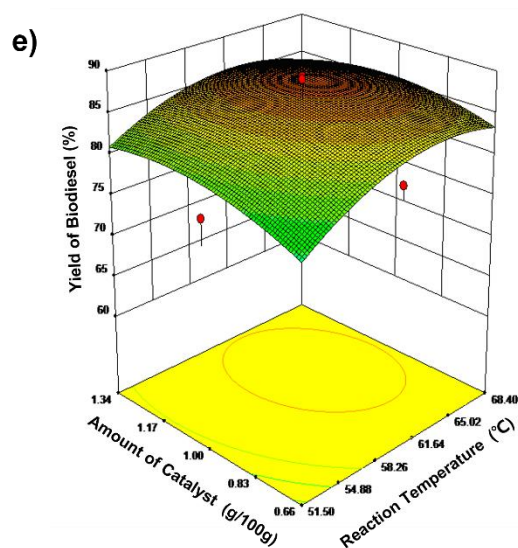
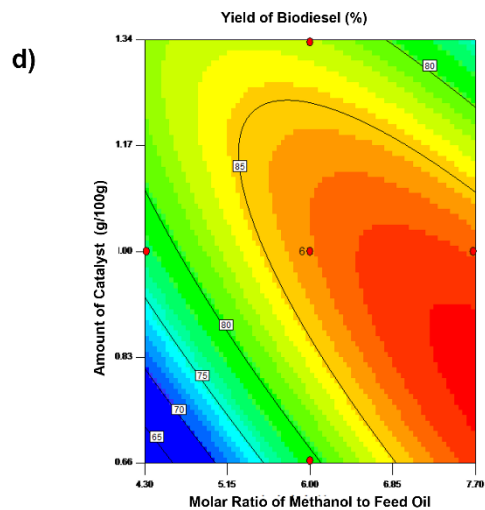


$$Y = f(X_1, X_2, X_3 = 1.0)$$

Fig. 4a) & b). (a, c, e) Response surface and (b, d, f) contour plots at various (a, b) reaction temperature (°C) and molar ratio of methanol to feed oil, (c, d) amount of catalyst (g/100 g) and molar ratio of methanol to feed oil, and (e, f) amount of catalyst (g/100 g) and reaction temperature (°C)



$$Y = f(X_1, X_3, X_2 = 60 \text{ } ^\circ\text{C})$$



$$Y = f(X_2, X_3, X_1 = 6:1)$$

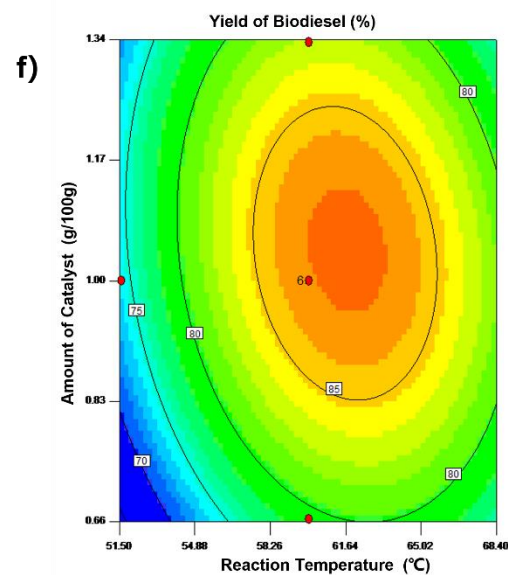


Fig. 4c) through 4f). (a, c, e) Response surface and (b, d, f) contour plots at various (a, b) reaction temperature ($^{\circ}\text{C}$) and molar ratio of methanol to feed oil, (c, d) amount of catalyst (g/100 g) and molar ratio of methanol to feed oil, and (e, f) amount of catalyst (g/100 g) and reaction temperature ($^{\circ}\text{C}$)

According to the shape of the contour line and the steepness of the response surface in Fig. 4 (Yuan *et al.* 2013), it can be judged that the interaction between alcohol oil molar ratio and NaOH dosage had a significant impact on the yield of biodiesel, while the interaction between alcohol oil molar ratio and reaction temperature, reaction temperature and NaOH dosage had no significant impact on the yield of biodiesel.

Validation of the best preparation technology of biodiesel

The test results were analyzed using Design expert software, and the preparation conditions were corrected with the best conditions: alcohol to oil molar ratio of 7:1, reaction temperature of 62 $^{\circ}\text{C}$ and NaOH dosage of 0.9%. Five parallel tests were

conducted in this condition, and the outcomes were 89.6%, 88.1%, 88.3%, 89.4%, and 90.0%, with an average of 89.0%, predicted value of 89.3%, and relative deviation of 0.30%, which proved that the regression equation has some applicability.

Determination of Fatty Acid Methyl Ester in *Xanthoceras sorbifolium* Oil-based Biodiesel

Determination of the linear relationship of the fatty acid methyl ester

Five mixed fatty acid methyl ester standard solutions with different concentrations were prepared using n-hexane as the solvent, and five different concentrations of methyl palmitate, methyl oleate, methyl linoleate, methyl linolenic acid, and methyl erucic acid were used as the fatty acid methyl ester. The substances of methyl oleate, methyl linolenic acid, methyl linoleate, and methyl erucic acid in 1 mL mixed solution were 2 mg, 4 mg, 6 mg, 8 mg, and 10 mg, respectively, and the content of methyl palmitate was 1 mg, 2 mg, 3 mg, 4 mg, and 5 mg, respectively. Results were obtained by taking 1 mL of 12 mg/mL methyl stearate, adding 0.25 mL to the above mixed standard solution, and making 0.75 mL of internal standard, mixing evenly, and then conducting gas chromatography analysis. Then the mass of each fatty acid methyl ester was divided by the mass of the internal standard as the horizontal coordinate (X-axis), and the peak area of each component of fatty acid methyl ester were divided by the peak area of the internal standard as the vertical coordinate (Y-axis). This approach was used to obtain the standard working curve of each fatty acid methyl ester. The results are shown in Table 6. The correlation coefficients (R) of each component were 0.9952, 0.9925, 0.9983, 0.9946, and 0.9988, separately, indicating that each fatty acid methyl ester had a good linear relationship in the corresponding concentration range.

Table 6. Standard Curve of Fatty Acid Methyl Esters

Standard Sample	Concentration Range (mg/mL)	Equation of Linear Regression
Methyl palmitate	0.75 to 3.75	$y = 3.7103x + 0.0298$
Methyl oleate	1.5 to 7.5	$y = 0.9030x - 0.1959$
Methyl linoleate	1.5 to 7.5	$y = 0.8417x - 0.0689$
Methyl linolenic acid ester	1.5 to 7.5	$y = 1.1285x - 0.1949$
Methyl erucate	1.5 to 7.5	$y = 0.2369x - 0.0026$

Table 7. Fatty Acid Methyl Esters Component of Biodiesel Made by *Xanthoceras sorbifolium* Seed Oil

Serial Number	Name	Molecular Formula	Content (mg/mL)
1	Methyl palmitate	$C_{17}H_{34}O_2$	57.15
2	Methyl oleate	$C_{19}H_{36}O_2$	291.84
3	Methyl linoleate	$C_{19}H_{34}O_2$	335.79
4	Methyl linolenic acid ester	$C_{19}H_{32}O_2$	71.58
5	Methyl erucate	$C_{23}H_{44}O_2$	68.36

Note: The density of *Xanthoceras sorbifolium* oil-based biodiesel is 0.8303 g/mL

Determination of fatty acid methyl ester in X. sorbifolium oil-based biodiesel

Under the above conditions, biodiesel was prepared from *Xanthoceras sorbifolium* Bunge oil and analyzed by gas chromatography under the same conditions. The test results are shown in Table 7. The table shows that the content of fatty acid methyl ester in *Xanthoceras sorbifolium* oil-based biodiesel was as high as 99.33%. Methyl esters containing 19 carbon atoms accounted for 84.8%, and hardly any S and N components. The results were perfectly consistent with the indicators of biodiesel.

CONCLUSIONS

1. Biodiesel was prepared from *Xanthoceras sorbifolium* Bunge seed oil with NaOH as catalyst, and the process was optimized using transesterification. The yield of biodiesel was 89.05% at 62 °C and 0.9% of the weight of catalyst oil.
2. *Xanthoceras sorbifolium* Bunge oil-based biodiesel was characterized by GC and quantitative analysis. It was determined that *X. sorbifolium* oil-based biodiesel was composed of methyl palmitate, methyl oleate, methyl linoleate, methyl linolenic acid, and methyl fatty acid methyl ester erucate, and the content of each fatty acid methyl ester was 99.33%, which is in line with the target of biodiesel. At the same time, such a composition is also an ideal product to replace fossil diesel.

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