Commercial CaO Catalyzed Biodiesel Production Process

Zaid Adnan Abdel-Rahman
Chemical Eng. Dept
Tikrit University
zaid572010@yahoo.com

Ahmed Daham Wiheeb
Chemical Eng. Dept
Tikrit University
ahmed_chem76@yahoo.com

Marwa Majeed Jumaa
Chemical Eng. Dept
Tikrit University
emaroosh@yahoo.com

Abstract

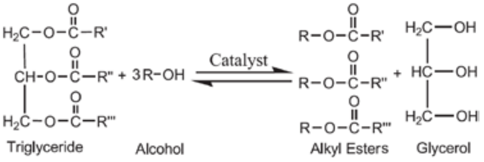
Biodiesel produced from vegetable oils is a good alternative clean diesel. The present study was conducted because there are some variations or contradictions in literature on the use of CaO heterogeneous catalyst. In this study, biodiesel was produced from sunflower vegetable oil and methanol in presence of commercial calcium oxide catalyst in batch mechanical stirrer reactor. The effect of three operating conditions, methanol mole ratio (4-12), reaction time (0.5-2.5 h) and catalyst amount (2-10 %), on the yield of biodiesel was studied at constant reaction temperature of 60 °C. Response surface methodology (RSM) was used with central composite design (CCD) of experiments. Polynomial correlation was found for the dependent variable of the process (yield of biodiesel), satisfactorily predicted at 95% confidence level. The optimum yield biodiesel was about 98% and at operating condition of methanol ratio 10, reaction time 2 h and catalyst amount 8 %. The reaction time was found to be the most effective operating condition. Kinetics study of the process showed that first order reaction with triglyceride concentration and zero order with methanol concentration gave best fit with the experimental data, triglyceride with a reaction rate constant k= 1.53 h^{-1}.

Keyword: CaO catalyst, Heterogeneous, Biodiesel Production, RSM

1. Introduction

Depletion of fossil fuel resources, caused by the heavy consumption, is a major global issue. The use of biodiesel, as a renewable source of energy, can help in solving this problem. Biodiesel is a good alternative fuel due to its characteristics like high degradability, no toxicity, and no sulfur emission [1].

Biodiesel is a mixture of alkyl esters and it can be produced by catalyzed transesterification reaction of vegetable oil (triglycerides) with excess methanol. The general equation for the transesterification reaction can be written as follows [1]:



(1)

Many types of alcohols can be used, e.g. methanol, ethanol, propanol and butanol. Methanol is preferred, because it is a relatively inexpensive alcohol and less consumption due to its low molecular weight [2]. The more general equation of reaction can be written as follows:

$$TG + 3\ MeOH \rightarrow 3\ FAME + GL \qquad (2)$$

Homogeneous basic catalyst, e.g. caustic soda (NaOH), process has many advantages; high activity, complete conversion within one hour, and mild reaction conditions, from 40 to 65°C and atmospheric pressure. The use of homogeneous catalyst leads to some disadvantages. The removal of the catalyst after reaction is technically difficult and a large amount of wastewater is produced. Heterogeneous calcium oxide catalyst eliminates these disadvantages and it is cheaper [1].

Peterson and Scarrach [3] firstly applied heterogeneous catalysts. Calcium oxide catalyst is less active than sodium hydroxide because their weak solubility in the reaction medium. The transesterification reaction rate in present CaO catalyst was found to be less than one half of that of NaOH catalyst. Table (1) shows the previous work dealing with CaO catalyst. There are some variations or contradictions in literature on the use of CaO heterogeneous catalyst. The range of the yield of biodiesel was between 50 to 99 % for approximately same process conditions. Also the reaction time varied from 1.5 to 10 h for biodiesel yield above 90% [4-35].

Table (1): Previous work of CaO catalyst

No.	Oil Type	Reaction Condition				Y %	Ref.
		C_{cat} %	R	T °C	t h		
1	Jatropha	1.5	9:1	70	2.5	93	[4]
2	Sunflower	1%	10	60	1.5	90	[5]
3	Sunflower	1%	12	50	5	95	[6]
4	Soybean	6.7	12	65	1 2	93 99	[7]
5	Soybean	8	12	65	3	80	[8]
6	Sunflower	10	6	60	1.5	98	[9]
7	Corn	1.5	9	65	4	98	[10]
8	Rapeseed	0.7	7	60	3	90	[11]
9	Soybean	1	13	65	2	99	[12]
10	Rapeseed	2	6	65	4	96	[13]
11	Palm	20	6	65	4	98	[14]
12	Sunflower	10	6	60	2 3	95 99	[15]

13	Palm	6	30	60	3	98	[16]
14	Sunflower	3	6	100	1.5	92	[17]
15	Soybean	14	9	70	10	90	[18]
16	Mustard	3	9	65	6	93	[19]
17	Mustard	3	9	60	3	95	[20]
18	Palm	4	7	60	4	95	[21]
19	Palm	10	12	60	3	90	[22]
20	Palm	10	9	65	>3	95	[23]
21	Jatropha	2.5	12	65	1	99	[24]
22	Pork lard	3	18	50	>4	94	[25]
23	Waste	6.7	10	60	1.7	95	[26]
24	Jatropha	1.5	6	60	1.5	99	[27]
25	Palm	5	9	60	3	60	[28]
26	Sunflower	5	12	60	2	95	[29]
27	Canola	3	9	65	2	99	[30]
28	Sunflower	3	12	60	3	95	[31]
29	Palm	4	12	65	3	50	[32]
30	Jatropha	4	15	65	6	90	[33]
31	Cotton	6	6	65	4	70	[34]
32	Jojoba	8	9	55 65	6.7	90	[35]

The objectives of the present study are to evaluate the use of commercial CaO heterogeneous catalyst for biodiesel production by designing and performing experiments for the effect of the operating conditions on the yield of biodiesel using response surface methodology (RSM), to obtain the optimum conditions of the process, and to study the kinetics of the reaction.

2. Experimental Work

Table (2) shows the physical properties of Turkish commercial edible sunflower oil used in the present study. Methanol of 98.99% purity (Thermo Huda Scientific/UK) was used. Fine commercial Iranian hydrated lime (purity>90%) was calcined at 900 ^{o}C for 2 hours under atmospheric pressure to obtain the active CaO catalyst as applied by Ibrahim et al [27].

Table (2): The physical properties of sunflower oil

Property	Value
Density (kg/m^3) at 15 ^{o}C	0.912
Kinematic viscosity (mm2/s) at 40 ^{o}C	33
Acid Value (mg KOH)	0.24
FFA %	0.085
Molecular weight (g/mole) [4]	883

Figure (1) shows methodology diagram for the transesterification process. The process consisted of 0.75 liter insulated stainless steel batch reactor placed on hotplate with temperature control. Figure (2) shows the batch stainless steel reactor. The reactor was of a 0.75 liter volume, diameter (D) of 100 mm, height (L) of 120 mm. The impeller was radial flat blade of 40 mm diameter. A 0.18 kW agitator was used with 600 rpm speed.

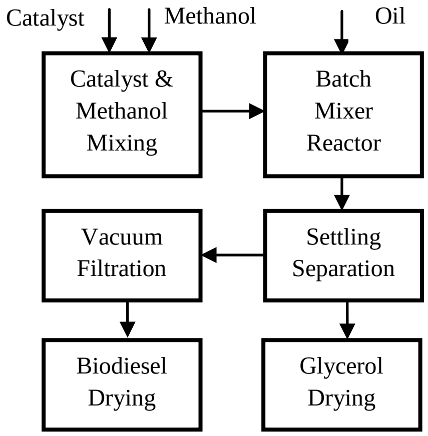


Figure (1): Methodology diagram

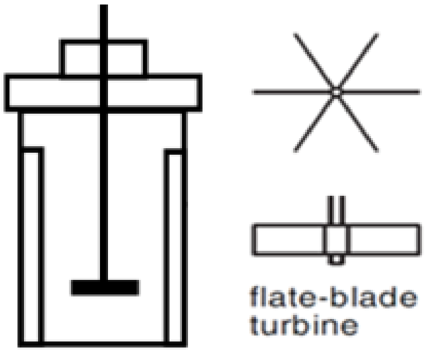


Figure (2): Batch stirrer reactor

The operating conditions ranges used in the present study were based on previous work literature [7] as follows;

- Alcohol mole ratio (R) =4-12
- Reaction time (t) = 0.5- 2.5 h
- CaO Catalyst (C_{cat}) = 2-10 %
- at constant reaction temperature T=60 ^{o}C

The experimental procedures was as follows;

1. Add 400 ml (364 g) of oil to the reactor and placed on hotplate until it reached the desired reaction temperature.
2. Mix a specified amounts of methanol and catalyst in conical flask.
3. Transfering the methanol and catalyst mixture to the reactor and mechanical stirring was started.
4. After a specified reaction time, the reaction mixture was transferd in 500 ml cylinder for settling overnight and some of the excess methanol was evaporated out.The reaction mixture was then separated into two layers by settling
5. The upper layer (biodiesel) was then vacuum filtrated by (buchner funnel and filtrer flask and filter paper ,Germany) and dryed (80˚C, 2 h) to remove the excess methanol and water.
6. The lower layer (Glycerol) was dryed (100˚C, 2 h) to remove the excess methanol and water, and weighting it.

7. The kinematic viscosity of biodiesel was measured by U-Tube viscometer in accordance with ASTM D-445.

8. The quality of glycerine produced was used for the calculation of the biodesiel yield (yield of glycerine equal to yield of ester), according to the general equation for the transesterification reaction, Eq. (2) to give the following equation;

$$Y = m_{GL} /(m_{TG}\, M_{GL}/M_{TG}) \qquad (3)$$

3. Experiments Design

In order to study the operating conditions for transesterification reaction and to investigate the interaction between the process variables, Design Expert 6.0.6 software was used, using a central composite design (CCD) of experiments. Three process variables; Methanol to oil ratio (R), reaction time (t), and catalyst amount (C_{cat}) were chosen with center values of 8, 1.5 h, and 6 % respectively. Center levels of the process variables were selected based on the ranges reported in literature for heterogeneous catalyzed transesterification process. Coded and actual process variables used in experiments design are presented in Table (2).

Table (2): Coded and actual process variables

Process variables	Coded and actual variables				
	-2	-1	0	1	2
Methanol ratio (R)	4	6	8	10	12
Time (t), h	0.5	1	1.5	2	2.5
Catalyst (C_{cat}), %	2	4	6	8	10

A 2^n full-factorial central composite design (CCD) for five levels of the three independent variables needs a total of 20 experiments according to 2^n+2n+6, where (n) is the number of independent variables. A second-order polynomial response surface model requires 20 experiments, as shown in Table (3). The center points are usually repeated 6 times to determine the experimental error and the reproducibility of the data [36].

Table (3): Experiments design (CCD), including results

Exp. No.	Variables						Yield %
	R	t, h	C_{cat} %	X_1	X_2	X_3	
1	6	1	4	-1	-1	-1	42
2	10	1	4	+1	-1	-1	53
3	6	2	4	-1	+1	-1	71
4	10	2	4	+1	+1	-1	85
5	6	1	8	-1	-1	+1	54
6	10	1	8	+1	-1	+1	69
7	6	2	8	-1	+1	+1	84
8	10	2	8	+1	+1	+1	95
9	4	1.5	6	-2	0	0	59
10	12	1.5	6	+2	0	0	81
11	8	0.5	6	0	-2	0	35
12	8	2.5	6	0	+2	0	92
13	8	1.5	2	0	0	-2	66
14	8	1.5	10	0	0	+2	85
15	8	1.5	6	0	0	0	79
16	8	1.5	6	0	0	0	81
17	8	1.5	6	0	0	0	78
18	8	1.5	6	0	0	0	83
19	8	1.5	6	0	0	0	82
20	8	1.5	6	0	0	0	77

4. Results and Discussion

The process of biodiesel production was analyzed by the application of the response surface methodology (RSM). Design Expert 6.0.6 software was used. A second-order polynomial model was obtained to predict the biodiesel yield as a function of three operating variables; alcohol to oil ratio (R=4-12), reaction time (t=0.5-2.5 h), and catalyst amount (C_{cat}=2-10%).

The analysis of variances (ANOVA), neglecting the insignificant terms (variables interactions, Rt, RC_{cat}, & tC_{cat}), resulted the empirical equation Eq. (4), with correlation coefficients and standard deviations of 0.9814 and 2.69%. Figure (3) shows a good correlation of the predicted verses actual biodiesel yield.

$$Y = -101.227 + 14.105\, R + 81.784\, t + 7.0085\, C_{cat} - 0.696\, R^2 - 17.636\, t^2 - 0.352\, C_{cat}^2 \qquad (4)$$

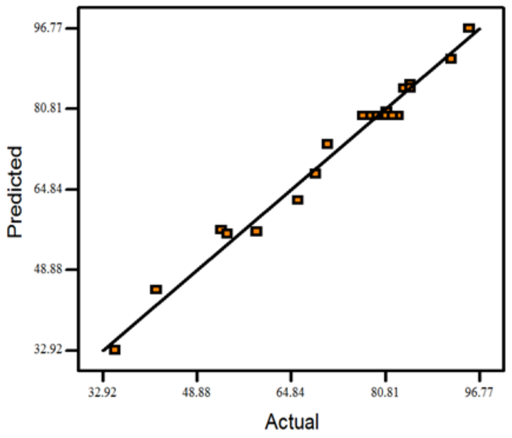


Figure (3): Predicted vs. actual biodiesel yield

Figure (4) shows the effect of the three operating variables (perturbation parameters), with reference center values; R=8, t=1.5 h, and C_{cat}=6 %, on the biodiesel yield. Reaction time was found to be the most effect variable. An increase of about 60 % in biodiesel yield was noticed on the range used of reaction time (t=0.5-2.5 h). Whereas an increase of about 20 % in biodiesel yield was noticed on the range used of both methanol mole ratio (R=4-12), and catalyst amount (C_{cat}=2-10%).

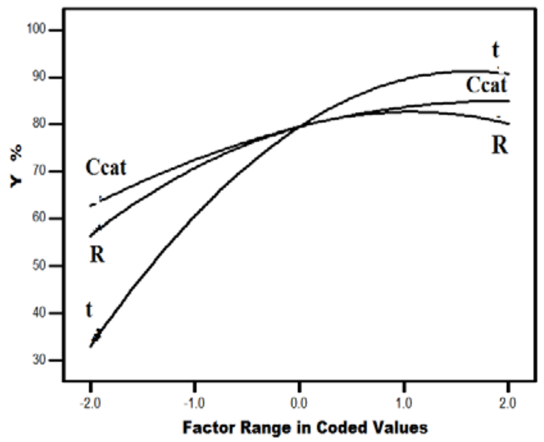
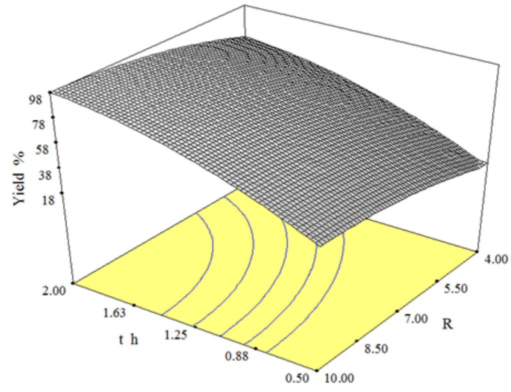
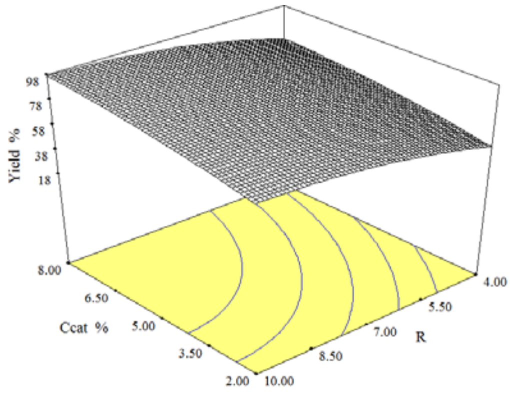


Figure (4): Effect of perturbation parameters on biodiesel yield

The optimum operating conditions was found, at R=10, t=2 h, and C_{cat}=8 %, to give a maximum biodiesel yield of about 98%, as shown in 3D Figure (5). Figure (5a) shows the effect of the two operating variables; alcohol mole ratio, and reaction time on biodiesel yield at an optimum value of catalyst amount. Whereas Figure (5b) shows the effect of the two operating variables; alcohol mole ratio and catalyst amount on biodiesel yield at an optimum value of reaction time. The present results in agreement of many literature results dealing with CaO catalyzed transesterification reaction of vegetable oil (sunflower) [9, 15, 17, 29, and 31]. Whereas they disagreement with others [5, 6]. This may be due to the activity and method of preparation of CaO catalyst. The variations or contradictions in literature on the use of CaO heterogeneous catalyst, as shown in Table (1) can be attributed to the difference in vegetable oil type and the activity and method of preparation of CaO catalyst.



(a) The effect of alcohol ratio and reaction time



(b) The effect of alcohol ratio and catalyst amount

Figure (5): The interaction effect of the three variables; alcohol ratio (R), reaction time (t), and catalyst amount (C_{cat}) on biodiesel yield at optimum conditions

Figure (6) shows the effect of reaction time on yield or conversion at optimum operating conditions; alcohol mole ratio (R=10), and catalyst amount (C_{cat}=8 %).

Assuming irreversible chemical reaction, the rate equation can be represented as follows [37]:

$$-r = k\, C_{GT}^{\alpha}\, C_{MeOH}^{\beta} \qquad (5)$$

First order reaction was found to give good fit (R^2=0.9647) with respect to the concentrations of triglyceride with a reaction rate constant k= 1.53 h^{-1}, as shown in Figure (7). Whereas second order reaction was found to give bad fit (R^2=0.7126), by plotting Y/(1-Y) versus time, Figure (8). This result is in agreement with that of Veljković et al., & Zhao et al [15, 30].

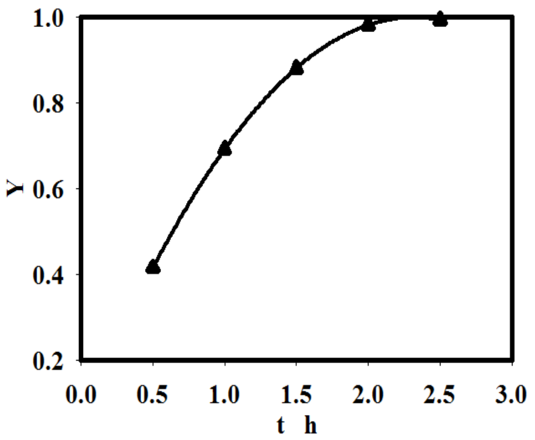


Figure (6): The effect of reaction time on yield or conversion at optimum operating conditions

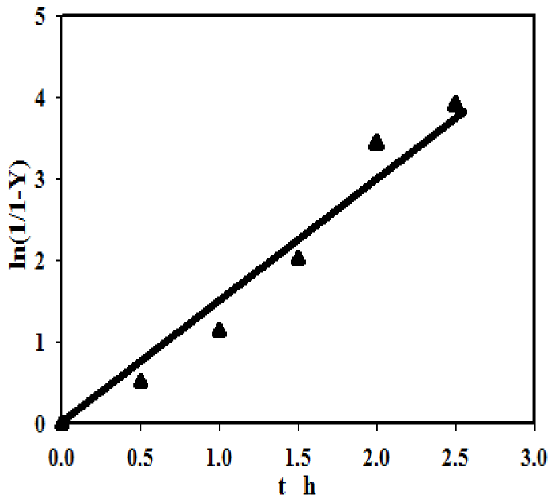


Figure (7): The first order behavior with respect to the concentrations of triglyceride

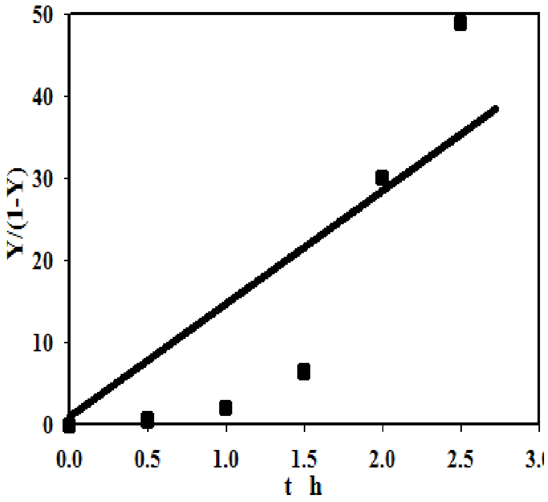


Figure (8): The second order behavior with respect to the concentrations of triglyceride

5. Conclusions

1. Commercial CaO heterogeneous catalyst was successfully applied for transesterification process.
2. Response surface methodology (RSM) was successfully applied for transesterification process, obtaining a second-order polynomial.
3. Reaction time was found to be the most effect process variable, 60% increase in biodiesel yield was noticed on the range used. Whereas an increase of about 20% in the yield was noticed on the range used for both methanol mole ratio, and catalyst amount.
4. The optimum operating conditions was found, at R=10, t=2 h, and Ccat=8 %, to give a maximum biodiesel yield of about 98%.
5. First order reaction with respect to the concentrations of triglyceride was found, with a reaction rate constant k= 1.53 h^{-1}.

Nomenclatures

B	Baffle width, mm
C_{cat}	Catalyst amount, %
C_{MeOH}	Concentrations of methanol, mole/l
C_{TG}	Concentrations of triglyceride, mole/l
D	Diameter of the reactor, mm
D_I	Diameter of impeller, mm
k	Reaction rate constant, h^{-1}
L	Height of the reactor, mm
M_{GL}	Glycerol molecular weight, g/mole
m_{GL}	Glycerol weight, g
M_{TG}	Oil (Triglyceride) molecular weight, g/mole
m_{TG}	Oil (Triglyceride) weight, g
n	Number of independent variables
r	Rate of reaction, mole/h
R	Methanol mole ratio
t	Time of reaction, h
X_1	coded variable of methanol mole ratio
X_1	coded variable to catalyst amount
X_2	coded variable of reaction time
Y	Yield of glycerine (equal to yield of ester)

Greek symbols

α	Order of reaction with respect to the concentrations of triglyceride
β	Order of reaction with respect to the concentrations of methanol

Abbreviations

ANOVA	Analysis of variances
CCD	Central composite design of experiments
FAME	Fatty acid methyl ether
FFA	Free fatty acid
GL	Glycerol
MeOH	Methanol
RSM	Response surface methodology
TG	Triglyceride

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عملية انتاج الديزل الحيوي باستخدام اوكسيد الكالسيوم التجاري كعامل المساعد

زيد عدنان عبدالرحمن
قسم الهندسة الكيمياوية
جامعة تكريت

احمد دحام وهيب
قسم الهندسة الكيمياوية
جامعة تكريت

مروة مجيد جمعة
قسم الهندسة الكيمياوية
جامعة تكريت

الخلاصة

البايوديزل المنتج من الزيوت النباتية خيار جيد لديزل نظيف . اجريت الدراسة الحالية لان هناك اختلافات او تناقضات في البحوث المنشورة على استعمال اوكسيد الكالسيوم كعامل مساعد غير متجانس. في هذه الدراسة , انتج البايوديزل من الزيت النباتي نوار الشمس مع الميثانول بوجود اوكسيد الكالسيوم التجاري كعامل مساعد في مفاعل دفعات بخلاط ميكانيكي. درس تاثير ثلاثة ظروف تشغيل ؛ نسبة المولية للميثانول (12-4) وزمن التفاعل (2,5 ساعة) وكمية العامل المساعد (2-10%) على انتاجية البايوديزل بثبوت درجة حرارة التفاعل 60 درجة مئوية. استخدمت طريقة استجابة السطوح (RSM) مع تصميم التجارب المركب المركزي (CCD) . تم ايجاد علاقة متعددة الحدود للمتغير المعتمد للعملية (انتاجية البايوديزل) , اعطت نتائج مقبولة في مستوى ثقة 95% . افضل انتاجية للبايوديزل بحدود 98% في ظروف تشغيلية ؛ النسبة المولية للميثانول (10) وزمن التفاعل (2 ساعة) وكمية العامل المساعد (8%). ووجد ان زمن التفاعل هو المتغير الاكثر تاثيرا لظروف التشغيل. بينت دراسة حركية العملية , ان تفاعل من الدرجة الاولى مع تركيز ثلاثي الكليسيرين ودرجة صفر مع تركيز الميثانول , اعطت افضل انطباق مع النتائج العملية , لثابت معدل التفاعل (k= 1.53 h^{-1}).