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Utilization of Electric Arc Furnace Dust as a Solid Catalyst in Biodiesel Production

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Research Article

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Abstract

World's energy sources like petrochemical oils, natural gas and coal cause global warming and environmental pollution. Therefore, the traditional energy sources must be replaced by the renewable energy resources. Biodiesel has been recognized as one of the effective, green, renewable and sustainable fuels. This paper investigates the production of biodiesel from sunflower oil by using electric arc furnace dust (EAFD) as a heterogeneous solid catalyst. Four reaction variables i.e. the reaction time, methanol to oil (M:O) molar ratio, reaction temperature, and EAFD loading were chosen to determine their effect on biodiesel production. The effect of the all reaction variables on the biodiesel yield was evaluated using response surface methodology (RSM). A relation has been developed representing the biodiesel conversion as function of all the independent variables. Reaction conditions optimization have been studied for the biodiesel yield maximization and the reaction conditions minimization. The optimum biodiesel yield equals 96 % at reaction temperature of 57 o C, Methanol to oil molar ratio of 20:1, and reaction time of 1h, and EAFD loading of 5%.

1. Introduction

Energy is the backbone of the world's life as there is no life on the earth without energy. Energy can define as the ability to do work or make change. Energy can be nonrenewable and renewable energy. Energy that comes from sources that will end or will need thousands or millions of years to form it called nonrenewable energy such as oil, coal and fossil fuels. Energy that can be easily produced and comes from resources like the wind and the sun are called renewable energy (Harrell, 2006). Solar energy, wind energy, hydropower, geothermal, and biofuels are examples for the renewable energy sources (Thangaraj, 2019). Biofuels are fuels that derived from biological carbon fixation as well as from biomass. In our world, there are many different types of biofuels such as bioethanol, biodiesel, bio-methanol, and biogas. Biodiesel and bioethanol are considered the most popular biofuels (Grassi, 1999).

Biodiesel was defined as mono alkyl ester of long chain fatty acids which obtained from renewable sources such as animal fats or vegetable oils. Biodiesel is used as an alternative fuel for diesel engines. In simple terms, Biodiesel is biofuel produced by chemical processes from animal fats or vegetable oils or recycled cooked oil and an alcohol that can be utilized alone or mixed with diesel oil in diesel engines. The prefix bio represents to biological and renewable source, while diesel represents to it utilize on diesel engines. The most common way to form biodiesel is through a process named "Trans-esterification" that involves using methanol to change the chemical properties of the oil. It is a simple process that produces high conversions with little percentage of glycerol as a byproduct (Hill et al., 2006).

The usage of catalyst in the trans-esterification reaction is very useful and important for higher yield of product and better rate of reaction. Catalysts utilized for trans-esterification of biodiesel feedstock may be heterogeneous or homogenous according to the solubility of the catalyst in the reaction (Chen et al., 2011). Many researches have been directed to the usage of heterogeneous catalysts for biodiesel-forming reactions to overcome the problems resulted when using homogenous catalysts. Although the homogenous acid catalyst is effectiveness, it can cause contamination issues which need product purification processes and good separation. This will increase the production process cost. Heterogeneous acid catalysts have many advantages such as regenerating and reusing catalyst (Amin Talebian-Kiakalaieh, 2013).

Electric arc furnace is considered the core equipment in steel manufacturing. The main function of such equipment is to carry out the melting processes for the given feed. This device produces a dangerous waste besides its main product. This waste is known as the electric arc furnace dust (EAFD). This material is known to be hazardous due to the high content of heavy metals mainly zinc and led. The phase of that waste is to be a solid waste basis. This material is known to have a degree of class 1 hazardous effect. The estimated amounts of waste are very large because it could reach from 15 kg to 25 kg of waste per a single tone of steel produced which could cause severe harmful for human and the environment (Rizescu & Stoian, 2011).

Kesic et al. (2012) used a mechano-chemically synthesized CaO·ZnO catalyst for biodiesel production. This catalyst gave biodiesel with yield approximately 99 wt% using methanol and sunflower oil (10:1 molar ratio) after 3 h reaction at 60 ^{o}C. Rengasamy et al. (2014) studied, the biodiesel production from castor oil using synthesized iron nanoparticles. The results of this study revealed that the produced biodiesel using synthesized iron nano-catalyst was considered as prospective alternate fuel to the conventional diesel fuel. Lukic et al. (2014) study the reaction kinetics of biodiesel production using ZnO supported on alumina/silica as a solid catalyst. The results proved the kinetics of the methanolysis reaction could be expressed by the first-order reversible reaction model. Al-Sakkari et al. (2016) studied the possibility of biodiesel production using cement kiln dust (CKD), as heterogeneous catalyst. The resulted optimum reaction conditions for biodiesel production were found to be 6 h reaction time, 2% catalyst loading, and 15:1 methanol to oil molar ratio at 65 °C reaction temperature and 800 rpm. Ahmed (2017) used zinc oxide nanoparticles as catalyst for biodiesel production by trans-esterification of waste cooking oil with yield 83%. Ali et al. (2017) prepared a nanocatalyst of CaO supported by Fe_3O_4 magnetic particles by a chemical precipitation method. The results revealed that the highest biodiesel yields for palm seed oil of 69.7% can be obtained under the conditions of (65 °C reaction temperature, 300 min reaction time, 20 methanol/oil molar ratio, and 10 wt.% of CaO/Fe_3O_4 catalyst loading).

The aim of this paper is to investigate the possibility of biodiesel production from sunflower oil using EAFD as heterogeneous catalyst and determining the optimum reaction conditions using response surface methodology (RSM).

2. Experimental Work

2.1 Raw Materials

The raw materials used in this work were as follow:

1. Electric arc furnace dust (EAFD) supplied from Egyptian steel factory at Ain El- Sokhna which was used to catalyze the reaction between sunflower oil and methanol.
2. Sunflower oil that was purchased from Egyptian local market used as liquid source in this work.
3. Methanol (MeOH) 99% that was supplied by Morgan Chemical Ltd., Egypt.

2.2 Assesment of EAFD

X–ray fluorescence is used for elemental analysis of EAFD to determine the concentration of various elements. X-ray diffraction (XRD) shows the phases present in the material. Standard sieving procedure is used to determine the particle size distribution as described by ASTM D 422 / 2007 was used. The sieves used are in compliance with ASTM E 11 / 2009. Scanning electron microscope with EDAX unit was used to determine the morphology of EAFD.

2.3 Biodiesel Production

The trans-esterification reaction took place in a round glass batch reactor fitted with reflux condenser. Sunflower oil was heated using magnetic heat stirrer then mixed with EAFD and methanol once the oil reached the targeted temperature. A reflux condenser was fitted at one of the reactor necks to condense the vaporized methanol during the reaction. Temperature was determined using a thermocouple. The mixture was filtered at the end the required reaction time using filter paper to remove the waste powder then transferred to a separating funnel after cooling to separate glycerol from biodiesel after 2 hours settling for sufficient separation of the products mixture.

The biodiesel was heated for 30 minutes at 80^{o}C for removal of unreacted methanol. The resulted biodiesel was weighted for conversion calculations by the following relation.

$$Biodiesel\ Conversion\% = \frac{Weight\ of\ biodiesel\ produced}{Weight\ of\ sunflower\ oil} x100 \quad (1)$$

2.4 Experimental Design

In this study, experimental design using response surface methodology (RSM) has been used to develop and construct a full analysis of the effect of reaction variables on reaction responses. Four independent variables have been selected which are reaction time, methanol to oil molar ratio (M:O), EAFD catalyst loading concentration and reaction temperature were named as A, B, C and D, respectively as shown in table (1). Biodiesel yield was selected as reaction responses.

Table 1. Factor affecting the trans-esterification reaction for biodiesel production

Factor	Label	Ranges	
		Minimum	Maximum
Reaction time (hr)	A	1	4
Methanol to oil molar ratio (M:O)	B	5	20
EAFD concentration (wt.%)	C	1	5
Reaction temperature (^{o}C)	D	50	70
Stirring Rate (rpm)	E	750	

Thirty experimental runs was done as an approach to minimize the number of experiments using central composite design technique (CCD) as shown in the table (2). The following table the experimental runs from 25 to 30 have the same conditions which called the centre point of design which means that performing a set of replicate tests at the same point. The proposed optimization targets have been selected based on economic and environmental considerations. Economic targets have been recognized for minimizing the cost of biodiesel production by minimizing the reaction conditions specially reaction time and temperature which means minimizing energy cost while maintaining maximum production of biodiesel from flower sun oil. Design Expert 12 software was used to investigate the experimental runs and its order, regression analysis, graphical analysis and numerical optimization. Eco-friendly or environmental biodiesel production was done using solid waste as solid heterogeneous catalyst which result in decreasing the production cost.

Table 2. Thirty runs generated by the design expert program

Run No.	Reaction Time, hr	Methanol/oil ratio	Catalyst loading, %	Temperature, °C
1	1	5	1	50
2	4	5	1	50
3	1	20	1	50
4	4	20	1	50
5	1	5	5	50
6	4	5	5	50
7	1	20	5	50
8	4	20	5	50
9	1	5	1	70
10	4	5	1	70
11	1	20	1	70
12	4	20	1	70
13	1	5	5	70
14	4	5	5	70
15	1	20	5	70
16	4	20	5	70
17	0.5	12.5	3	60
18	5.5	12.5	3	60
19	2.5	3	3	60
20	2.5	27.5	3	60
21	2.5	12.5	0.5	60
22	2.5	12.5	7	60
23	2.5	12.5	3	40
24	2.5	12.5	3	80
25 - 30	2.5	12.5	3	60

2.5 Experimental Work Done on Optimum Biodiesel Sample

Physicochemical properties were determined for the optimum biodiesel sample and compared to Biodiesel International Standards ASTM D6751 and the European Biodiesel Standard, EN14214. The final optimum biodiesel sample derived from sunflower oil has been analyzed through gas chromatography. Analysis of total FAME and methyl linoleate in produced biodiesel has been conducted according to the standard EN 14103 method. In addition, analysis of total and free glycerol and triglycerides content in B100 biodiesel according to the standard EN14105 method.

2.6 Reusability of EAFD

The optimum biodiesel sample which was resulted from using M:O molar ratio of 20:1, catalyst loading of 5%, reaction temperature of 57^{o}C, reaction time of 1 h and stirring rate of 750 rpm which gave 96% for biodiesel conversion was undergone filtration for solid particles removal then this solid particles were washed with methanol for glycerol removal then finally dried to remove any traces of methanol on the particles. This EAFD was reused to catalyze trans-esterification reaction of sunflower oil under the same optimum reaction conditions and without adding any new solid catalyst to reaction medium. After conducting trans-esterification reaction, the conversion was calculated for each experiment to test the catalyst efficiency and strength.

3. Results And Discussion

3.1 Analysis of EAFD

3.1.1 Chemical Analysis of EAFD

Table (3) shows the results of X-ray florescence (XRF) analysis for EAFD. The solid waste consists mainly of Fe_2O_3, ZnO, CaO and SiO_2 oxides with other oxides. These oxides are good biodiesel catalyst according to previous research work so this is a good indication that this solid waste can be used as biodiesel catalyst.

Table 3. Chemical analysis of EAFD

Oxides	%
Fe_2O_3	32.2
ZnO	23.1
CaO	19.4
SiO_2	5.9
Al_2O_3	0.6
MnO	1.1
MgO	0.9
Na_2O	2.3
K_2O	3.42
SO_3	2.5
TiO_2	0.2
P_2O_5	0.02
Cl	2.6
L.O.I.	5.8

3.1.2 Mineralogical Analysis of EAFD

The mineralogical analysis of the waste powder in figure (1) shows that it mainly consists of zinc oxide, zinc iron oxide, sodium manganese silicate hydroxide and potassium manganese sulfide phases.

3.1.3 Screen Analysis of EAFD

Fig. (2) shows the cumulative screen analysis curve of EAFD waste. The vertical axis represents the fraction retained on each screen with certain diameter. This figure shows that the grind waste is very fine. The mean particle size of waste powder equal 6.93643 μm

3.1.4 Analysis on Produced Biodiesel:

The biodiesel was generated and its conversion was calculated and table (4) shows the results of each experimental run. Regression equation was generated using Design Expert software. Analysis of variance (ANOVA) was used to investigate the significance of the resulted model at 95% confidence level so the significance level equals 5% which means that the P-values must be less than 0.05. Design expert program can calculate the critical F values for each model by knowing the number of variables, number of samples, and significant level and then compare the values of F for model with the critical one to know if the model is significant or not. F-test and p-value was used to determine the significance of each parameter and validate the response surface methodology (RSM) model. F-test and p-value equals 62.47 and less than 0.0001, respectively so the resulted model was statistically significant. The design expert program suggest the quadratic model to be the best model using ANOVA analysis technique. Some terms in the quadratic model were neglected as they are insignificant as their P-values are greater than 0.1 so the model become a reduced quadratic model. Figure (3) shows the results of ANOVA analysis that was done by the design expert program. The following equation represents the relationship betweenthebiodiesel conversion andthereactionconditions as a reduced quadratic model.

$$Y = 1.135\,A + 3.682\,B + 0.623\,C + 4.407\,D - 0.033\,BD - 0.036B^2 - 0.028\,D^2 - 91.392 \qquad (3)$$

Where Y is the biodiesel conversion or the reaction response, A is the reaction time in hrs, B is methanol to oil ratio, C is Catalyst loading as wt. %, and D is the reaction temperature in ^oC.

Coefficient values, R^2_{adj} and R^2 were determined to measure the validity of the fitting model which was found to be 0.9369 and 0.9521 and, respectively which insure the high significanceofthepredictedmodel.

The predicted values were plotted versus experimental results of the biodiesel conversion. This plot shows reasonable agreement and high correlation as shown in Figure (4).

Table 4. Biodiesel conversion at each experimental run

Run No.	Biodiesel Conversion,%	Run No.	Biodiesel Conversion,%
1	70.2	14	97.8
2	74.9	15	98.5
3	84.8	16	99.7
4	95.4	17	93.9
5	74.9	18	95.0
6	78.5	19	78.3
7	90.4	20	99.9
8	92.1	21	93.9
9	85.5	22	95.0
10	93.2	23	68.1
11	97.9	24	97.4
12	98.6	25 - 30	94.3
13	93.2		

3.1.4.1 Effect of Each Reaction Condition on Biodiesel Conversion

Fig. (5) Shows the effect of Methanol/oil molar ratio, reaction time, reaction temperature, and catalyst loading on the biodiesel conversion. The reaction temperature and M:O ratio have the highest effect of the biodiesel conversion while the catalyst loading and reaction time have a very small effect on the biodiesel conversion.

3.1.4.2 Reaction Variables Interactions Effect on Biodiesel Conversion

The following surface plots represent the effect of reaction variables interactions on biodiesel conversion. Fig. (6) Shows the contour plot that represents the relation between M:O ratio and temperature interactions (BD) and biodiesel conversion while Fig. (7) shows its surface or 3D plot. Fig. (6) and Fig. (7) show that the combination of M:O ratio and temperature has a big effect on the biodiesel conversion. Fig. (8) shows the surface plot that represents the relation between effect Methanol/oil and reaction time on biodiesel conversion and indicates that the combination between Methanol/oil and reaction time have a little effect on the biodiesel conversion compared with the combination of M:O ratio and temperature.

3.1.5 Optimization of Reaction Variables

The Design expert software developed optimum 100 solutions within the required targets and the optimum solution was the solution with the highest desirability (74 %). The optimum conditions using numerical optimization have been concluded at M:O molar ratio of 20:1, catalyst loading of 5%, reaction temperature of 57°C, reaction time of 1 h and stirring rate of 750 rpm resulting in 96% for biodiesel conversion. Figure (9) shows the summary for the optimization constrains with some resulted solutions by the program showing the desirability of each solution.

3.1.6 Analysis on Optimum Produced Biodiesel Sample:

Table (5) shows the physicochemical properties and its standard limits. All of the properties agree with both EN14214 and ASTM D 6751.

Table 5: Physicochemical properties of the resulted optimum biodiesel sample

Physicochemical Properties	**Results**	**Standard Method**	**EN14214**	**ASTM D6751**
Calorific value (MJ/kg)	40.83	ASTM D-5865	> 32.9	
Pour point (°C)	-22	ASTM D-97		
Cloud point (°C)	-14	ASTM D-97	< -4	
Flash point (°C)	170	ASTM D-93	> 101	> 130
Kinematic viscosity at 40°C (cSt)	4.8	ASTM D-445	3.5 – 5.0	1.9 – 6.0
Density at 15°C (g/cm^3)	0.89	ASTM D-4052	0.86 – 0.9	

The final optimum biodiesel sample derived from sunflower oil has been analyzed through. Table (6) illustrates the results concluded from Gas Chromatography (GC) analyses for the optimum produced sample. It is clearly shown that the produced biodiesel has agreed with the specified specification for both European standards EN 14103 and EN 14105. Both free glycerol and total glycerol in the produced biodiesel samples did not exceed the specification limits of EN 14105 by recording 0.01 and 0.019 % (m/m), respectively. Similarly, monoglycerides, diglycerides and triglycerides concentration in the final pure biodiesel recorded acceptable results in comparison with EN 14105 by recording 0.0043, 0.0078, 0.0856 % (w/w), respectively. Finally, total FAME concentration has recorded using EN 14103, It has an acceptable result with concentration of 97.6 %. (m/m).

Table 6: Gas chromatography results

Test	Results	Specification	
		Min	Max
Free Glycerol	0.01 %		0.02 %
Total Glycerol	0.019 %		0.25 %
Monoglycerides	0.0043 %		0.80 %
Diglycerides	0.0078 %		0.20 %
Triglycerides	0.0856 %		0.20 %
Total FAME	97.6 %	96.5 %	

3.1.7 Reusability Results of EAFD:

Figure (10) shows that the conversion dropped from 96% after the first use and reached 85% at after the second run then decreased to about 70% by the end of third use and finally decrease to 50% at the end of fourth use. There are many reasons for decreasing the activity of the catalyst such as the contamination of glycerol on the active center of the catalyst. The second reason is leaching of catalyst which means active species loss from the solid because of its transformation into the liquid medium. The results showed that EAFD can be reused only two or three times as a maximum after that a new catalyst will be used.

4. Conclusion

An economic biodiesel production from sunflower oil has been examined using electric arc furnace dust (EAFD) solid waste as a heterogeneous catalyst. The XRF analysis shows that EAFD solid waste consists mainly of Fe_2O_3, ZnO, CaO and SiO_2 oxides so this is a good indication that this solid waste can be used as biodiesel catalyst. Four independent reactions conditions i.e. the reaction temperature, methanol to oil (M:O) molar ratio, reaction time, and catalyst loading have been chosen to detect their effect on biodiesel production. Thirty experimental runs was done as an approach to minimize the number of experiments. The effect of all reaction variables on the biodiesel yield was evaluated using response surface methodology (RSM). A model has been generated representing the biodiesel conversion as function of all independent variables. The M:O ratio and reaction temperature have the highest effect while the catalyst loading and reaction time have a very small effect on the biodiesel conversion. The Design expert software has developed optimum 100 solutions within the required targets which is minimizing the biodiesel production cost and maximization its produced amount. The optimum conditions using numerical optimization have been concluded at M:O molar ratio of 20:1, catalyst loading of 5%, reaction temperature of 57^{o}C, reaction time of 1 h and stirring rate of 750 rpm resulting in 96% for biodiesel conversion. Gas chromatography analysis showed that the produced biodiesel has agreed with the specified specification for both European standards EN 14103 and EN 14105. The reusability proved that EAFD can be reused only two or three times as a maximum after that a new catalyst will be used.

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Figures

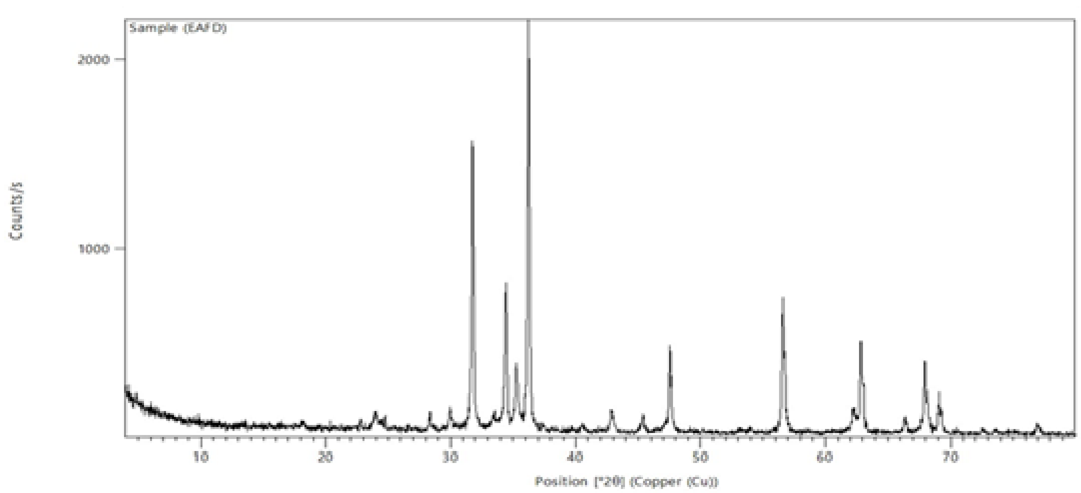

Figure 1

Mineralogical analysis of EAFD

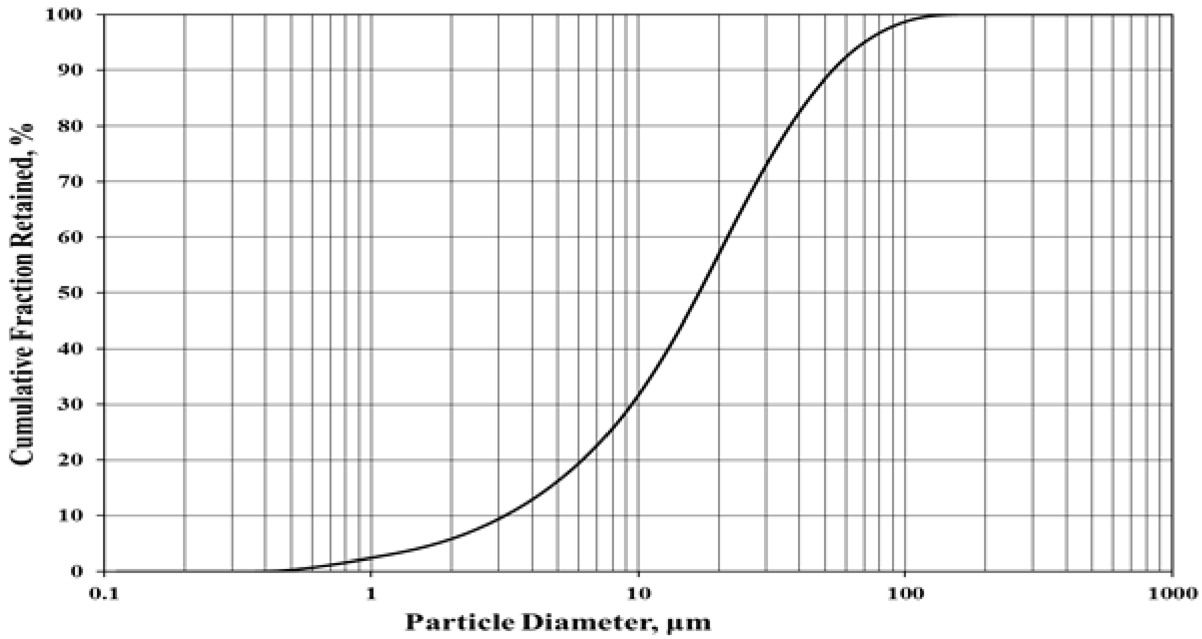

Figure 2

Cumulative screen analysis curve of EAFD waste

Analysis of Variance

ANOVA for Reduced Quadratic model

Response 1: Biodiesel Conversion

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2284.10	7	326.30	62.47	< 0.0001	significant
A-Reaction Time	63.01	1	63.01	12.06	0.0022	
B-Methanol/Oil ratio	758.80	1	758.80	145.27	< 0.0001	
C-Catalyst Loading	33.40	1	33.40	6.39	0.0191	
D-Temperature	1089.91	1	1089.91	208.66	< 0.0001	
BD	96.34	1	96.34	18.44	0.0003	
B^2	71.71	1	71.71	13.73	0.0012	
D^2	220.04	1	220.04	42.13	< 0.0001	
Residual	114.91	22	5.22			
Lack of Fit	114.91	17	6.76			
Pure Error	0.0000	5	0.0000			
Cor Total	2399.01	29				

Figure 3

Results of ANOVA analysis

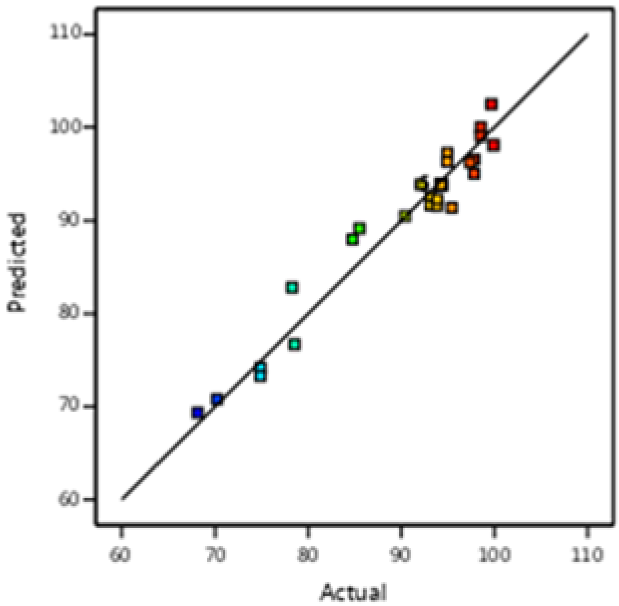

Figure 4

Relation between the predicted and experimental results of the biodiesel yield

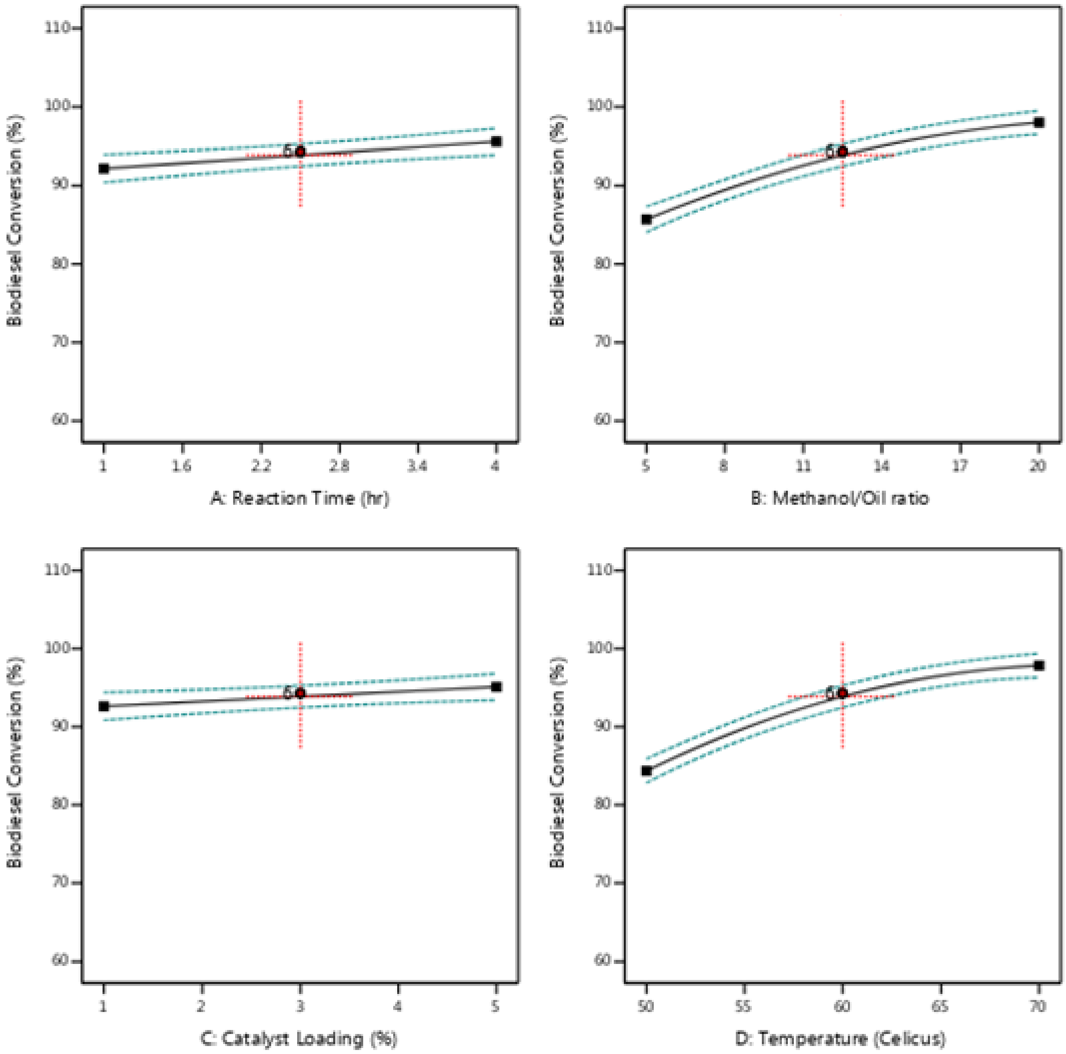

Figure 5

Effect of different process variables on the biodiesel yield

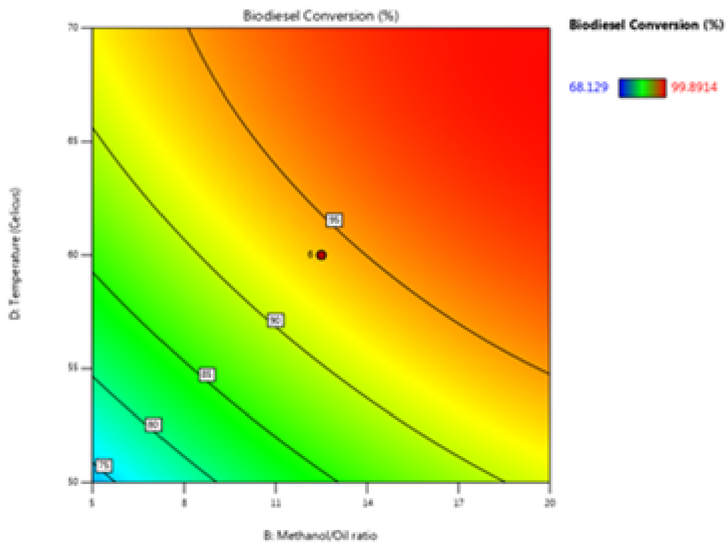

Figure 6

Contour plot for the relation between M:O ratio and temperature interactions and biodiesel conversion.

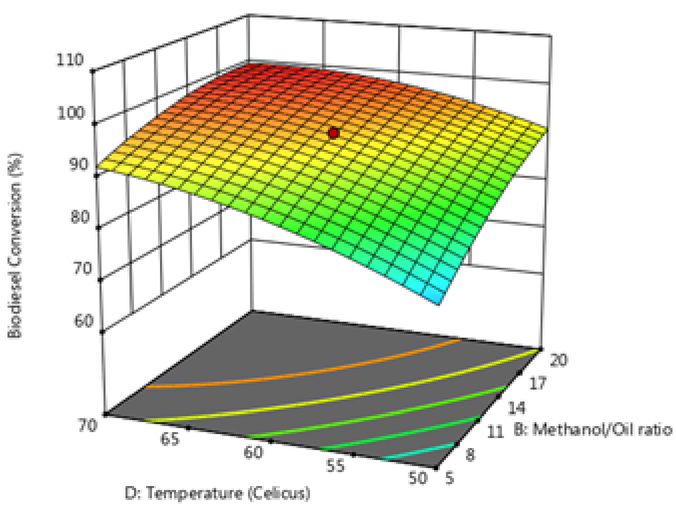

Figure 7

Surface plot show the relation between M:O and reaction temperature and biodiesel conversion.

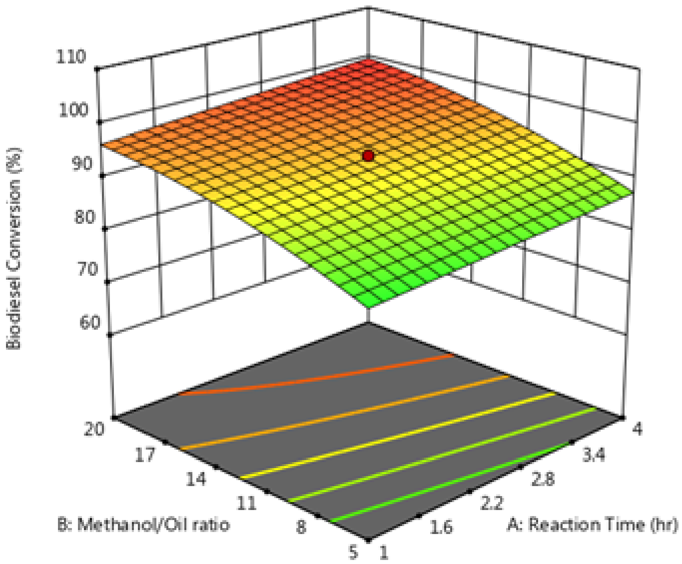

Figure 8

Surface plot show the relation between effect methanol/oil and reaction time on biodiesel conversion.

Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Reaction Time	minimize	1	4	1	1	5
B:Methanol/Oil ratio	is in range	5	20	1	1	3
C:Catalyst Loading	is in range	1	5	1	1	3
D:Temperature	minimize	50	70	1	1	5
Biodiesel Conversion	maximize	68.129	99.8914	3.63078	1	5

Solutions | Starting Points

Solutions

100 Solutions found

Number	Reaction Time	Methanol/Oil ratio	Catalyst Loading	Temperature	Biodiesel Conversion	Desirability	
1	**1.000**	**20.000**	**5.000**	**57.023**	**96.034**	**0.740**	**Selected**
2	1.000	19.997	5.000	56.937	95.981	0.740	
3	1.000	20.000	4.999	56.736	95.861	0.740	
4	1.000	20.000	4.984	57.007	96.015	0.740	
5	1.000	19.995	5.000	56.657	95.811	0.740	
6	1.000	20.000	4.981	57.384	96.232	0.740	
7	1.016	20.000	5.000	57.059	96.074	0.739	

Figure 9

Optimization constrains and results.

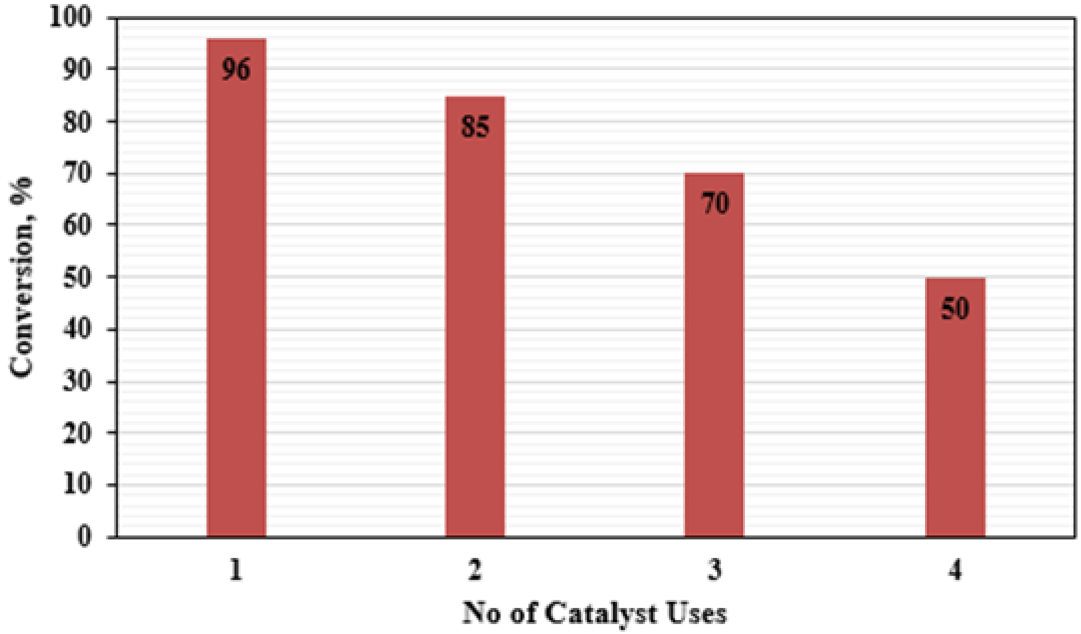

Figure 10

Reusability test results