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Process optimization for biodiesel production from waste frying oil with constructional lime

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Abstract

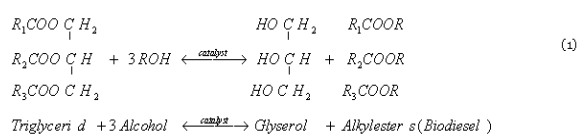
Lime as a cheap and abundant constructional material has shown good activity for catalyzing biodiesel production from waste frying oil. In the present work, due to lack of data for optimum operating conditions and interaction effects, an optimization study was performed using response surface methodology. The effects of catalyst weight percent, MeOH/Oil molar ratio and calcination temperature in relation to oil conversion as response were also investigated and optimized. Using Box-Behnken design and quadratic polynomial model, the optimum level for catalyst weight percent, MeOH/Oil molar ratio and calcinations temperature were determined as 3 wt%, 13.1, and 716.2 °C, respectively. At these conditions, the anticipated oil conversion was 92.3%. Catalyst weight percent and calcination temperature are two parameters that mainly affect the oil conversion and a significant interaction can be observed between them.

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Introduction

Biodiesel is a sustainable diesel-equivalent fuel that can be produced from renewable raw materials and its negative environmental effect is negligible in comparison to fossil diesel fuel. Biodiesel is consisting of long-chain fatty acid alkyl esters made from vegetable oils, recycled cooking greases, or animal fats and has similar physical and chemical properties with petro-diesel fuel. Moreover, it has beneficial advantages over fossil fuels including non-toxicity, biodegradability, significantly lower emissions, low sulfur and aromatic content (Demirbas, 2008; Ghanei *et al.*, 2011; Helwani *et al.*, 2009). Biodiesel is prepared from transesterification of different vegetable oil or animal fat with a short chain alcohol, in which one mole triglyceride reacts with three moles methanol in the presence of appropriate amount of catalyst to form three moles of mono methyl ester and one mole of glycerol. This reaction is shown in the following equation (1) (Balat and Balat, 2010; Demirbas, 2009; Ghanei *et al.*, 2011; Helwani *et al.*, 2009; Moradi *et al.*, 2012).

Different types of edible and non-edible vegetable oils have been exploited in the past and present to produce biodiesel with promising results. The used oils include cotton seed, soybean, peanut, palm, waste cooking, rapeseed, sunflower seed, canola seed, winter rape, frying, jojoba, karanja, tobacco seed and jatropha curcas (Balat and Balat, 2010; Boey *et al.*, 2011; Demirbas, 2009; Ghanei *et al.*, 2011; Ghanei *et al.*, 2013; Helwani *et al.*, 2009; Leung *et al.*, 2010; Moradi *et al.*, 2012; Wang *et al.* 2012). The cost of raw materials has a major effect on the total cost of biodiesel production and thus choosing waste oils can be a promising alternative to the other expensive oils (Balat and Balat, 2010; Boey *et al.*, 2011; Ghanei *et al.*, 2013; Leung *et al.*, 2010; Wang *et al.* 2012).



Generally, enzymes, strong acids and bases are used as a catalyst in two forms; homogeneous and heterogeneous (Boey *et al.*, 2011; Demirbas, 2009; Ghanei *et al.*, 2011; Ghanei *et al.*, 2013). Homogenous

bases are able to complete a reaction successfully, however the catalyst recovery is rather difficult and large amounts of hazardous waste water can be formed during these reactions. On the other hand, heterogeneous catalysts can be easily separated from products and also can be used several time without any waste water formation (Boey *et al.*, 2011; Ghanei *et al.*, 2013; Helwani *et al.*, 2009; Leung *et al.*, 2010). This type of catalysts allows researchers to design continuous process namely fixed bed reactors (Boro *et al.*, 2012). Moreover, solid acids such as I_2/ZnO , SO_4^{2-}/ZrO_2 , SO_4^{2-}/TiO_2 , carbon-based solid acid catalyst, carbohydrate-derived catalyst, Vanadyl phosphate, niobic acid, sulphated zirconia, Amberlyst-15, Nafion-NR50 and solid bases like CaO , $CaTiO_3$, TiO_2-MgO , $CaMgO$, $CaZnO$, KF/Eu_2O_3 , KF/ZnO , $CaZrO_3$, $CaO-CeO_2$, $CaMnO_3$, $Ca_2Fe_2O_5$, KOH/Al_2O_3 , KOH/NaY , KI/Al_2O_3 , ETS-10 zeolite, alumina/silica supported K_2CO_3 were also investigated (Boro *et al.*, 2012; Helwani *et al.*, 2009; Leung *et al.*, 2010; Refaat, 2011). Among the mentioned catalysts, calcium oxide is the most well-accepted and excellent heterogeneous catalyst (Albuquerque *et al.*, 2008; Boey *et al.*, 2009; Boey *et al.*, 2011a; Boey *et al.*, 2011b; Boro *et al.*, 2012; Demirbas, 2009; Ghanei *et al.*, 2013; Kouzu *et al.* 2008; Liu *et al.*, 2008; Nakatani *et al.*, 2009; Refaat, 2011; Viriya-empikul *et al.*, 2010; Viriya-empikul *et al.*, 2012). The main influential parameters of transesterification are as follows: molar ratio of alcohol to oil, catalyst amount, reaction temperature and pressure, catalyst calcinations temperature, mixing intensity, reaction time and the contents of FFAs and water in oil (Balat and Balat, 2010; Boey *et al.*, 2009; Boey *et al.*, 2011a; Boey *et al.*, 2011b; Demirbas, 2009; Hameed *et al.*, 2009; Jeong *et al.*, 2009; Kandedo *et al.*, 2009; Sahoo and Das, 2009; Vicente *et al.*, 2007a; Vicente *et al.*, 2007b; Zabeti *et al.*, 2009). Vicente *et al.* (2007a; 2007b) have been investigated the effect of reaction temperature, methanol/oil molar ratio and catalyst concentration on the product yield by means of response surface method in transesterification of sunflower oil in the presence of KOH. Additionally, Gwi-Taek Jeong *et al.* (2009) have been used response surface methodology

to optimize the temperature, methanol/oil molar ratio and catalyst concentration in biodiesel production from lard using KOH as a catalyst. Patil and Deng (2009) have been optimized the operating conditions including reaction temperature, reaction time, methanol/oil molar ratio and catalyst concentration for transesterification of jatropha, karanja, corn, and canola oil in the presence of KOH and H₂SO₄ as catalysts. Sahoo and Das (2009) have also investigated the reaction parameters of biodiesel production from jatropha, karanja, and polanga oil using KOH as a catalyst. Wang *et al.* (2012) have studied the optimum reaction conditions for transesterification of *Datura stramonium* L. oil using KOH and H₂SO₄ as catalysts. In most cases, optimum reaction temperature was close to the boiling point of alcohol, optimum catalyst concentration was between 0.5 to 1 wt% of oil weight and methanol/oil molar ratio was around 6 for alkali catalyzed reactions (Balat and Balat, 2010; Demirbas, 2009; Jeong *et al.*, 2009; Leung *et al.*, 2010; Patil and Deng, 2009; Sahoo and Das, 2009; Vicente *et al.*, 2007a; Vicente *et al.*, 2007b; Wang *et al.* 2012). In the case of heterogeneous catalysis, Hameed *et al.* (2009) have found the optimum methanol/oil molar ratio, catalyst wt%, and reaction time are equal to 11.43, 5.52%, and 9.72 h, respectively, using response surface method for the transesterification of palm oil and using KF/ZnO as a catalyst. Zabeti *et al.* (2009) have performed an optimization study based on response surface method for biodiesel production from palm oil using CaO/Al₂O₃ as catalyst and introduced the optimum condition for loading the amount of CaO on the support. The amount of loaded CaO on the catalyst and the calcination temperature were equal to 100.54 wt% and 718°C, respectively. Kansedo *et al.* (2009) have been studied the transesterification of palm oil via a heterogeneous process using montmorillonite KSF as catalyst based on response surface methodology with four variables including reaction temperature, reaction time, methanol/oil molar ratio, the amount of catalyst, and optimum condition. The obtained values are as follows: reaction temperature 190°C, reaction time 180 min, methanol/oil molar ratio 8:1, and the amount of

catalyst 3%. Boey *et al.* (2009) have been investigated the optimum conditions for transesterification of palm oil in the presence of calcined crab shell and the reported values are as follows: reaction temperature 65°C, methanol/oil mass ratio 0.5, catalyst amount 5%, and mixing rate 500 rpm. In another study, Boey *et al.* (2011a) have determined the optimum condition using response surface methodology for transesterification of palm oil in the presence of calcined cockle shell and reported the obtained values as follows: methanol/oil mass ratio 0.54, and catalyst amount 4.9%.

Biodiesel, as a promising source, can be prepared from waste frying oil in the presence of heterogeneous catalysts. Such reaction needs to be optimized. Among all the operating conditions, methanol/oil molar ratio, catalyst amount and calcinations temperature have a major effect on the yield of reaction (Boey *et al.*, 2011a; Hameed *et al.*, 2009; Zabeti *et al.*, 2009). In our previous work, constructional lime was selected for its availability and abundance to produce biodiesel from waste frying oil (Ghanei *et al.* 2013). According to our investigations, calcinations temperature is one of main influential parameters which also show high interaction with other factors. As a result, in the present work optimization study with response surface methodology was performed for three factors (catalyst amount, methanol: oil molar ratio, and calcinations temperature) at three different levels, to produce biodiesel from waste frying oil in the presence of constructional lime as heterogeneous catalyst.

Materials and methods

Waste frying oil (WFO) was collected from local restaurant. Constructional lime was supplied from construction workshop in campus. All reagents were used are listed below: methanol 99.9% (chromatography grade, Merck), potassium hydroxide 85% pellets (GR for analysis, Merck), Hydrochloric Acid 37% (GR for analysis, Merck), n-Hexane 95% (extra pure, Merck) and some other chemicals were purchased from local chemical

companies. Methyl Laurate (Methyl Dodecanoate) \geq 99.7% were supplied from Sigma as a standard for GC analysis.

Catalyst pretreatment

Subsequent to the lime powder sieving, excess amount of lime with particle sizes between 180 μ m to 250 μ m was charged into a crucible and then it was put in an electrical furnace. The furnace was then heated up to the assigned temperature and heated for 4 hrs. Then the activated catalyst was cooled to 150°C and appropriate amount of catalyst weighed and transferred to a reactor, immediately.

Catalyst Characterization

The surface area and pore size of the constructional lime which was preheated at 600°C was determined through N₂ adsorption/desorption using a Quantachrome NOVA Automated Gas Sorption System (Quantachrome Instruments). Prior to the N₂ adsorption/desorption measurement, the catalyst was heated under nitrogen flow at 573 K overnight in a Quantachrome Flow Degasser to remove any impurities that might have adsorbed on the solid during its exposure to air. The X-ray diffraction (XRD) patterns of the constructional lime at different calcination temperatures were also recorded on a Bruker D8 Advance X-Ray Diffractometer equipped with a graphite monochromator, copper radiation, and scintillation counter (detector) in the range of $2\theta = 5-80^\circ$.

Waste frying oil pretreatment

Since the collected waste frying oil was dark and contained solid particles, it was heated up to melt the solid part of the oil and then WFO was filtered under vacuum to remove the solid impurity. The filtered oil was then heated up to 110°C for 15 min in order to evaporate the water (Sabudak and Yildiz, 2010). Soap index was determined for the waste frying oil according to the Cd 3b-76 AOCS official methods (1998). Soap value plays an important role in determining the average molecular weight of oil. Acid value was also determined for the waste frying oil

according to the Ca 5a-40 AOCS official methods (1998).

Biodiesel synthesis

Transesterification of waste frying oil was carried out in a 250 ml three neck flat bottom flask equipped with a condensation and temperature controlling system. The reactor was then placed on a hot plate magnetic stirrer. The operational conditions were as follows: atmospheric pressure; reaction temperature: 65°C; mixing rate: 1000 rpm; and reaction time: 5 h. Variable conditions were as follows: weight of catalyst: 0.5, 3, and 5.5% which is based on the initial oil weight; methanol/oil molar ratio: 6, 12, and 18; calcinations temperature: 300, 600, and 900°C. The reactor was first charged by 100 g of oil and then heated up to the required reaction temperature. Then, the mentioned amount of catalyst was added to the oil. Finally, methanol was added to reactor and the reaction was initiated. The flask was cooled immediately at the end of the reaction and reaction mixture was filtered to remove the catalyst. Then produced biodiesel was washed with hot distilled water until the drainage and distilled water pH became the same. At the end, the product heated at 110°C for 15 min to eliminate residual water and methanol.

Biodiesel characterization

The composition and quantity of fatty acid methyl ester (FAME) in biodiesel was determined using a HP 6890 Gas Chromatograph with a flame ionization detector FID. The capillary column was a BPX-70 high polar column with a length of 120 m, a film thickness of 0.25 μ m and an internal diameter of 0.25 mm. Nitrogen was used as carrier gas and also as an auxiliary gas for the FID. One micro-liter of sample was injected using a 6890 Agilent Series Injector. Lauric acid methyl ester (methyl laurat) was added as a reference into the crude biodiesel and the samples were analyzed by the above mentioned GC. The analysis of biodiesel for each sample was carried out according to Ghanei *et al.* (2011).

$$FAME \text{ wt}\% = \left(\frac{\text{area of all FAME}}{\text{area of reference}} \times \frac{\text{weight of reference}}{\text{weight of Biodiesel sample}} \times \frac{f_{FAME}}{f_r} \right) \times 100 \quad (2)$$

Where f is correction factor for transforming area percentage to weight percentage that was obtained from calibration.

Oil characterization

Saponification index, free fatty acid wt% and fatty acid composition of waste frying oil after initial pretreatment were measured and reported in Table 1.

Results and discussion

Table 1. Obtained properties and fatty acid composition for waste frying oil.

<i>Characterization</i>	
Free Fatty Acid (wt%)	2.17
Saponification index (mg KOH/g oil)	207.67
Mean Molecular Weight (g/mol)	810.416
<i>Fatty Acid Composition (wt%)</i>	
Lauric (C12:0)	0.35
Myristic (C14:0)	0.85
Palmitic (C16:0)	30.14
Palmitoleic (C16:1)	3.15
Margaric (C17:0)	---
Margaroleic (C17:1)	---
Stearic (C18:0)	5.65
Oleic (C18:1)	41.21
Linoleic (C18:2)	17.54
Linolenic (C18:3)	0.75
Arachidic (C20:0)	---
Eicosenoic (C20:1)	---
Behenic (C22:0)	0.35
Erucic (C22:1)	---

Table 2. Surface area and pore size data for the calcined lime at 600°C

<i>Catalyst name</i>	<i>BET area(m²/g)</i>	<i>surface BJH area(m²/g)</i>	<i>Surface BJH (cm³/g)</i>	<i>Pore volume BJH (Å)</i>	<i>Pore diameter</i>
calcined lime at 600°C	6.064	6.921	3.792e-2	25.3	

Table 3. Experimental design matrix and results.

Run Order	Level of variables (actual)			Level of variables (coded)			Conversion (%)
	Cat. wt%	MeOH/oil molar ratio	Calcination Temp.(°C)	A	B	C	
1	5.5	12	300	+1	0	-1	54.6
2	3.0	12	600	0	0	0	81.5
3	0.5	6	600	-1	-1	0	51.0
4	5.5	18	600	+1	+1	0	94.8
5	3.0	12	600	0	0	0	91.0
6	0.5	18	600	-1	+1	0	58.9
7	5.5	12	900	+1	0	+1	44.6
8	0.5	12	300	-1	0	-1	2.0
9	3.0	12	600	0	0	0	89.4
10	3.0	6	300	0	-1	-1	18.5
11	3.0	6	900	0	-1	+1	75.9
12	3.0	18	300	0	+1	-1	22.5
13	5.5	6	600	+1	-1	0	85.5
14	3.0	18	900	0	+1	+1	78.0
15	0.5	12	900	-1	0	+1	93.4

Catalyst characterization

The calcined constructional lime at the temperature of 600°C was first analyzed according to section 2.2 and then the surface area, pore volume and pore diameter presented in Table 2. According to the reported results, surface area and pore volume of the mentioned catalyst are not acceptable. Medium pore diameter put this catalyst in mesoporous category.

XRD patterns for constructional lime with different calcination temperatures are presented in Figure 1. According to the results, constructional lime with no calcination is originally composed of calcium carbonate (~80%) and calcium hydroxide (~20%). In this step no CaO phase has been detected. Also during the calcination at 300°C, no significant phase change was observed. Subsequent to the calcination process

at 600°C, calcium carbonate (in form of aragonite) and calcium hydroxide peaks disappeared completely, and calcium oxide peaks were appeared. Calcination at higher temperature contributes to transformation of all compounds to CaO. Since the active component in transesterification of triglycerides is calcium oxide, thus constructional lime without calcination cannot catalyze the reaction. Therefore, a pretreatment at a temperature of 600°C is required to regenerate the CaO which is transformed to Ca(OH)₂ during the storage time, due to water absorption. At this stage, unconverted CaCO₃ plays as a support for the catalyst. Additionally, a preheating at the temperature of 900°C is required to convert all constituents to CaO as reported by Liu *et al.* (2008) and Kouzu *et al.* (2008).

Table 4. ANOVA values for conversion.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	11821.3	11821.31	1313.48	8.81	0.014
Linear	3	5475.1	5475.13	1825.04	12.24	0.010
Square	3	3774.3	3774.30	1258.10	8.44	0.021
Interaction	3	2571.9	2571.88	857.29	5.75	0.045
Residual Error	5	745.6	745.62	149.12		
Lack of fit	3	693.9	693.88	231.29	8.94	0.102
Pure Error	2	51.7	51.74	25.87		
Total	14	12566.9				

Optimization study

In order to perform an optimization study, a Design of Experiment (DOE) was used. An unblocked Box-Behnken Design with three factors including catalyst wt%: 0.5-5.5% which is based on the initial oil weight; MeOH/oil molar ratio: 6-18; calcinations

temperature: 300-900°C. The mentioned design has 15 run experiments with 3 center points and reaction conversion (%) was chosen as a response. The experimental design matrix and results are presented in Table 3.

Table 5. Optimization condition and results.

Criteria	Goal	Lower limit	Upper limit	Optimum condition
Reaction Conversion (%)	Maximum	0	100	92.274
Catalyst weight percent	In range	0.5	5.5	3.0
MeOH/Oil molar ratio	In range	6	18	13.122
Calcination temperature (°C)	In range	300	900	716.244

All reactions were performed according to section 2.4, and the reported conditions in Table 3. Moreover, the

product conversions were measured according to section 2.5. The obtained values have been reported

in Table 3. According to the other investigations, quadratic model can be selected as an appropriate model for statistical analysis and optimization (Boey *et al.*, 2009; Boey *et al.*, 2011a; Boey *et al.*, 2011b; Hameed *et al.*, 2009). Furthermore, from the Analysis of Variance (ANOVA) (Tables 4), the “Prob > F” value for the quadratic model was significant (<0.05). Values less than 0.1000 indicated that the model terms are significant. The F value for response was quite high; giving more emphasis to the variance that was explained by the model. In addition, the p-values of the studied variables were found to be less than 0.05, implying their significant effects on the responses.

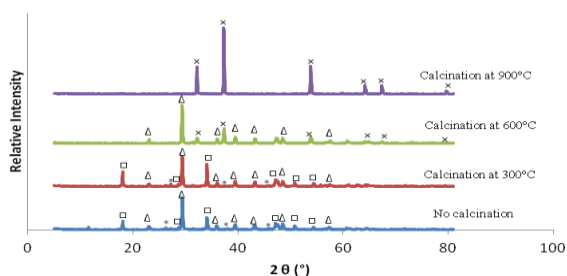


Fig. 1. XRD patterns of constructional line at different calcination temperatures. (× : CaO , □ :Ca(OH)₂, Δ : CaCO₃(calcite) , * : CaCO₃(aragonite).

The final empirical model in terms of coded factor (Y) is shown in Eq. (3).

$$Y = 87.3 + 9.28A + 2.91B + 24.29C - 7.41A^2 - 7.34B^2 - 31.24C^2 + 0.35AB - 25.35AC - 0.48BC \quad (3)$$

Where “Y” is oil conversion (%) and “A”, “B”, and “C” are catalyst weight percent, MeOH/Oil molar ratio, and calcinations temperature (°C), respectively.

The terms with positive sign have a synergistic effect, whereas the terms with negative sign have an antagonistic effect. The quality of the developed model was evaluated based on the correlation coefficient value (Hameed *et al.*, 2009). A model with a closer R² value to the unity, provides predicted values which are closer to the actual values for the response. The R² of 0.941 for Eq. (2) was considered relatively high, indicating the fact that there is an agreement between the actual and predicted conversion of waste frying oil to biodiesel from this

model. Fig. 2 shows the predicted values for reaction conversion according to Eq. 3. The data closeness to the unit slope line in the Fig.2 for quadratic model implies that this model is the best model to represent the factors affecting biodiesel conversion.

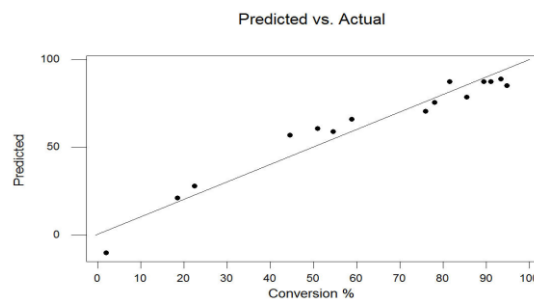


Fig. 2. Predicted values; conversion versus actual values (solid line is y=x).

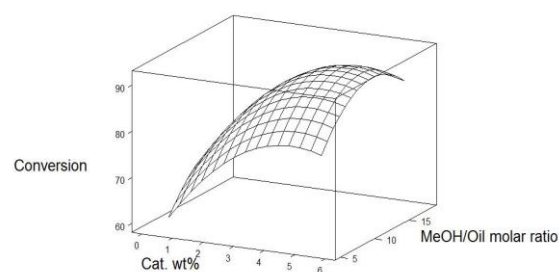


Fig. 3. Response surface plot ; waste frying oil conversion versus catalyst weight percent and MeOH/Oil molar ratio (Calcination temperature: 600°C).

Figures 3, 4, and 5 show the three-dimensional response surfaces demonstrating the effects of variables on the reaction conversion. Fig. 3 shows the effect of catalyst weight percent and MeOH/Oil molar ratio on conversion (calcination temperature: 600°C). Fig. 4 shows the effect of MeOH/Oil molar ratio and calcination temperature on conversion (catalyst weight percent: 3%). Fig. 5 shows the effect of catalyst weight percent and calcination temperature on conversion (MeOH/Oil molar ratio: 12). According to the obtained values from eq.2 (figures 3-5), catalyst weight percent and calcinations temperature are the most influential factors and MeOH/Oil molar ratio is the least influential factor in relation to conversion. All mentioned variables have an optimum point which can be affected by other variables. This means that the interaction effects between the variables are significant. Catalyst weight percent has a positive

influence on the conversion when calcination temperature is low. Such situation is reversed in the case of high temperatures of calcination (Figure 5). MeOH/Oil molar ratio is not highly affected by other factors. Fig. 6 illustrates the main effect of all variables on response (conversion) and Fig. 7 shows the interaction between variables.

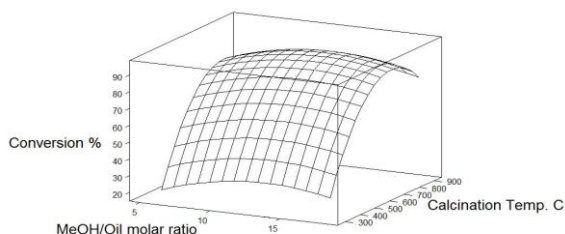


Fig. 4. Response surface plot; waste frying oil conversion versus MeOH/Oil molar ratio and calcination temperature (Catalyst weight percent: 3%).

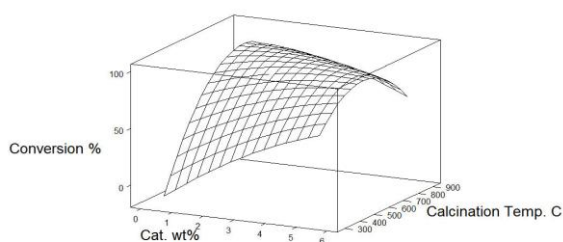


Fig. 5. Response surface plot; waste frying oil conversion versus catalyst weight percent and calcination temperature (MeOH/Oil molar ratio: 12).

The highest yield was obtained when all three variables were at the optimum point within the studied range. Therefore a numerical method was selected to optimize the response using a Minitab® response optimizer. The most desirable value was equal to 0.92367 which was obtained when catalyst weight percent, MeOH/Oil molar ratio, and calcination temperature were 3.0 %, 13.122, and 716.224°C, respectively. At these conditions waste frying oil conversion was 92.274%. Table 5 summarizes the optimization condition and results. Boey *et al.* (2009) have used calcined Mud Crab Shell as a catalyst to transesterify palm olein and have been noticed that the optimal values of reaction temperature, catalyst weight percent, MeOH/Oil mass ratio, and stirring rate are equal to 65°C, 5 wt%,

0.5 (~ 13 molar ratio), and 500 rpm, respectively. In another investigation, Boey *et al.* (2011a) have determined the optimal catalyst weight percent and MeOH/Oil mass ratio for biodiesel production from palm olein in the presence of calcined cockle shell as catalyst which was equal to 4.9 wt% and 0.54 (~14 molar ratio). Nakatani *et al.* (2009) have also reported the catalyst weight percent of 25wt.% and 5h reaction time for the transesterification of soybean oil catalyzed by combusted oyster shell. Zabeti *et al.* (2009) have reported the temperature of 718°C as the optimum calcination temperature for CaO/Al₂O₃ in transesterification of palm oil.

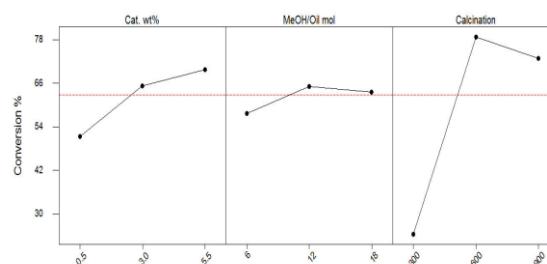


Fig. 6. Main effect plots for all variables of waste frying oil conversion.

Conclusion

In order to verify the optimal reaction condition for biodiesel production from waste frying oil in the presence of constructional lime as a catalyst, Box-Behnken Design with three factors including catalyst wt%: 0.5-5.5% based on initial oil weight; MeOH/oil molar ratio: 6-18; calcinations temperature: 300-900°C and waste frying oil conversion as response, were considered. Fixed operation conditions were as follows: atmospheric pressure; reaction temperature: 65°C; mixing rate: 1000 rpm; and reaction time: 5h. Using response surface methodology with quadratic polynomial model optimum level for catalyst weight percent, MeOH/Oil molar ratio and calcinations temperature were obtained as 3 wt%, 13.1, and 716.2 °C, respectively. At these conditions, predicted conversion of oil was 92.3%. Among these factors catalyst weight percent and calcination temperature are the most influential factors and MeOH/Oil molar ratio is the least influential factor in relation to conversion. Also interaction effects between the factors were significant.

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