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ORIGINAL PAPER

Utilization of waste coral for biodiesel production via transesterification of soybean oil

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Abstract In the present study, the waste coral was utilized as a source of calcium oxide for transesterification of soybean oil into biodiesel (methyl esters). Characterization results revealed that the main component of the waste coral is calcium carbonate which transformed into calcium oxide when calcined above 700 °C. The Box–Behnken design of experiment was carried out, and the results were analyzed using response surface methodology. Calcination temperature, methanol– soybean oil molar ratio and catalyst concentration were chosen as variables. The methyl ester content (wt%) was response which must be maximized. A second-order model was obtained to predict methyl ester content as a function of these variables. Each variable was placed in the three low, medium and high levels (calcination temperature of 700, 800 and 900 °C; catalyst concentration of 3, 6 and 9 wt%; methanol-to-oil ratios of 12:1, 18:1 and 24:1). The optimum conditions from the experiment were found that the calcination temperature of 900 °C, catalyst concentration of 6 wt% and methanol-to-oil ratio of 12:1. Under these conditions, methyl ester content reached to 100 wt%. The waste catalyst was capable of being reused up to 4 times without much loss in the activity.

Keywords Coral · Biodiesel · Transesterification · Calcium oxide

Introduction

Due to reduction in fossil resource and increasing environmental concerns, researchers have been concentrated on alternative fuels such as bioethanol and biodiesel, in recent years (Balat 2011; Demirbas 2007; Pedroso et al. 2006; Semwal et al. 2011). The most common technology of biodiesel production is transesterification of oils (triglycerides) with alcohol which gives biodiesel (fatty acid methyl esters, FAME) as main product and glycerol as by product (Boey et al. 2011). Different kinds of vegetable oils and animal fats were used as triglyceride resource by many researchers (Goodrum 2002; Guru et al. 2010; Jeong et al. 2009; Zhang et al. 2003; Refaat 2010). Biodiesel can be used neat or mixed with petroleum diesel to produce a biodiesel blend that can be used in ignition engines (Demirbas 2009). There is no need to modify the engine or system when it is operated with biodiesel (Cheung et al. 2009). Actually, transesterification cause to decrease viscosity of triglyceride and prepared biodiesel for using in engine (Ngamcharussrivichai et al. 2010).

Vegetable oils can be transesterified by heating them with an alcohol and a catalyst. Catalysts used in biodiesel production are divided in two general categories, homogenous and heterogeneous types. In this time, the biodiesel industry is dominated by application of homogeneous catalysts (such as KOH, NaOH and CH3ONa) because of their easy usage and short time required for conversion of triglycerides to biodiesel (Demirbas 2008; Sharma et al. 2011). However, the use of homogeneous catalysts requires neutralization and separation from the final reaction products leading to a series of environmental concerns such as production of waste water and corrosion problems (Georgogianni et al. 2009).

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Heterogeneous or solid catalysts are more beneficial simple separation and reuse (Di Serio et al. 2006). Among heterogeneous catalysts which have been researched, calcium oxide shows high potential in transesterification reaction. This catalyst is low cost, low methanol solubility, non-corrosive, environmental friendly and able to be reused for several times (Boey et al. 2011).

CaO was used as a solid base catalyst for transesterification of soybean oil to biodiesel by (Lui et al. 2008). The experimental results showed that 12:1 molar ratio of methanol to oil, addition of 8 wt% CaO catalyst based on the weight of oil, 65 °C reaction temperature gave the best results and biodiesel yield exceeded 95 % after 3 h. Biodiesel was produced from sunflower oil using calcium oxide by Granados et al. (2007). Powder of CaO was calcined at 1,000 °C. Conversion was obtained 94 % at 60 °C reaction temperature for 100 min, 13:1 molar ratio of methanol to oil and 3 wt% catalyst content. CaO was obtained after calcination of pulverized lime stone in a helium gas flow at 900 °C for 1.5 h by Kouzu et al. (2008). CaO was calcined at 550 °C by Veljkovic et al. (2009). After 2 h and 12:1 molar ratio of methanol to soybean oil, yield of biodiesel production exceeded 93 %.

Dolomite stone was calcined as a source of CaO at 800 °C by Ngamcharussrivichai et al. (2010). The experimental conditions were 6 wt% catalyst content, the methanol–oil molar ratio of 30:1 and reaction time was 3 h. Methyl ester content was achieved to 98 %.

Waste shells as source of CaO have been used by some researcher. Waste eggshell was used as low-cost solid catalyst for biodiesel production by Wei et al. (2009). Waste eggshell was calcined at 1,000 °C, and it caused that $CaCO_3$ transformed into CaO. Biodiesel yield reached to 97–99 % at 65 °C reaction temperature, 9:1 molar ratio of methanol to oil and 3 wt% catalyst content. Combusted waste oyster shell was used as a catalyst for transesterification of soybean oil by Nakatani et al. (2009). The catalyst was calcined at 700 °C. The biodiesel purity was obtained 94.8 % at 5 h with 6:1 molar ratio of methanol to oil and 25 wt% catalyst content. Waste mud crab (*Scylla serrata*) shell was used as a heterogeneous catalyst for biodiesel production via transesterification of palm olein (Boey et al. 2009a, b). $CaCO_3$ transformed into CaO when the shell was calcined above 700 °C for 2 h. The optimal conditions were found to be methanol–oil mass ratio 0.5:1, catalyst amount of 5 wt%, reaction temperature of 65 °C and a stirring rate of 500 rpm. Waste shells of mollusk and egg were used as catalysts for producing biodiesel by Viriya-empikul et al. (2010). The shells were calcined in air at 800 °C for 4 h. Biodiesel yield was obtained 90 % after 2 h at 60 °C reaction temperature, 18:1 molar ratio of methanol to oil and 10 wt% catalyst content.

In the present study, the powder of waste coral was used as a source of CaO for transesterification of soybean oil. Dead or waste corals have been found at the beach of Persian Gulf enormously. The effects of calcination temperature, molar ratio of methanol to soybean oil and catalyst concentration on methyl ester content were investigated. In addition, the reusability of catalyst was investigated. The experimental results were analyzed using response surface methodology.

Materials and methods

Materials

Soybean oil was obtained from Nazgol Oil Company (Kermanshah, Iran), and waste coral was obtained from the coast of Persian Gulf, south of Iran.

Methanol (for synthesis, >99.5 %) and standard methyl ester for using in gas chromatography column were purchased from Merck.

Preparation of waste coral as catalyst

The waste corals were roughly crushed using a hammer, and after that powder of coral was sieved (<1 mm). The powder was calcined by air in a furnace at 700, 800, 900 °C for 2.5 h.

Experimental procedure

Transesterification reaction was conducted with different catalyst concentrations, molar ratios of alcohol to oil (methanol: oil) and calcination temperatures. All experiments were done within a 250-ml-two-necked flask that equipped with a thermometer and a condenser. Soybean oil, methanol and powder of catalyst were added to the two-necked flask. Then the mixture was stirred for 8 h, and the temperature was kept at 65 °C using an oil bath. After the reaction, the catalyst was separated by centrifugation, and then, glycerol and produced biodiesel were separated within a separation funnel. Then, to increase the purity, the produced biodiesel was washed several times by 90 °C distilled water; finally, to remove the water, it was placed in the oven with the temperature of 100 °C for 3 h.

The calcined corals were characterized by X-ray diffraction (XRD). A PW-1800 Philips X-ray diffractometer with monochromatized CuK_α radiation ($\lambda = 1.5406$ Å) was used for X-ray measurement. Data were collected over a 2θ range of 5–80°, with a step size of 0.02° at a scanning speed of 1 min^{-1}.

BET surface area, pore volume and pore diameter were measured by N_2 physisorption at 77 K using NOVA 2000 Series (Quantachrome, USA). Prior to the adsorption–desorption measurements, all the samples were degassed at 150 °C in N_2 flow for 16 h. Results were obtained using a BET equation at $P/P_0 = 0.9918$.

The elemental compositions were determined by energy dispersive X-ray fluorescence spectroscopy. A PW-1800 Philips X-ray fluorescence has been used for elemental analyzing. XRF analysis which used for this test was not able to determine the elements which their composition below 1 wt%.

Determination of methyl ester content

Characterization of fatty acid methyl ester (FAME) in biodiesel samples was determined by 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 µm and an internal diameter of 0.25 mm. Nitrogen was used as the carrier gas and also as an auxiliary gas for FID. One microliter of the sample was injected using a 6890 Agilent Series Injector with a splitless mode. The inlet temperature of sample into injector was 50 °C, which was heated up to 230 °C at a heating rate 5 °C/min. Methyl laurate (C12:0) was added as a reference into the crude biodiesel, and the samples were analyzed by GC that was mentioned above.

The analysis of biodiesel for each sample was done according to Wang et al. (2006). Methyl ester content wt% was calculated using Eq. (1) as follows:

$$\text{Methyl ester content (wt\%)} = \frac{(\sum A)}{A_s} \times \frac{W_s}{W} \times 100 \quad (1)$$

where $\sum A$ sum of all areas under the curve from C12 to C24, A_s area under the curve of C12:0 (Reference), W_s Weight of C12:0 (g) (Reference) and W Weight of biodiesel sample (g).

Statistical design of experimental

In this study, the Box–Behnken experimental design technique is used to achieve optimum methyl ester content of biodiesel production. The experimental ranges and levels of the variables of transesterification process are listed in Table 1. The effects of calcination temperature (T), catalyst concentration (C) and molar ratio of methanol to oil (MR) on methyl ester content were investigated. Each variable was considered in three levels, the bottom (−1), medium (0) and high (+1). Calcination temperature was investigated at three levels: 700, 800 and 900 °C; catalyst concentration was investigated in three levels: 3, 6 and 9 wt%; and molar ratio of methanol to oil was investigated at three levels: 12:1, 18:1 and 24:1. The total number of trails was 15 that included 13 trials with two repetitions at the center point for identifying the errors. Box–Behnken experimental design and the results of the tests are given in Table 2. Methyl ester content (Y) of produced biodiesel is the responses to the experimental design. Second-order model for these responses are presented in Eq. (2):

$$Y = \beta_0 + \beta_1 T + \beta_2 C + \beta_3 \mathrm{MR} + \beta_4 T^2 + \beta_5 C^2 + \beta_6 \mathrm{MR}^2 + \beta_7 \mathrm{TC} + \beta_8 \mathrm{TMR} + \beta_9 \mathrm{CMR} \quad (2)$$

In which Y is the projected response (methyl ester content); T, C and MR are the input variables (calcination

Table 1 Range of changes and the level of operational variables

Factors	Symbol	Range and levels		
		−1	0	+1
Calcination temperature, °C	T	700	800	900
Catalyst concentration, wt %	C	3	6	9
Molar ratio of methanol to oil	MR	12:1	18:1	24:1

Table 2 Experimental design for three independent variables and the response

Run	Manipulated variables			Response
	Calcination temperature (°C)	Catalyst concentration (wt %)	Molar ratio of methanol to oil	Methyl ester content (wt %)
1	700	3	18	44.3
2	700	6	12	47.22
3	700	6	24	41.62
4	700	9	18	45.75
5	800	3	12	78.66
6	800	3	24	71.96
7	800	6	18	83.12
8	800	6	18	83.64
9	800	6	18	82.96
10	800	9	12	77.45
11	800	9	24	73.02
12	900	3	18	93.16
13	900	6	12	100
14	900	6	24	90.64
15	900	9	18	93.62

temperature, catalyst concentration and molar ratio of methanol to oil, respectively); β_0 is the intercept; β_1, β_2 and β_3 are linear coefficients; β_4, β_5 and β_6 are second-order model coefficients; and β_7, β_8 and β_9 are the interaction coefficients between variables.

Mathematical model experiment is tested via variance analysis (ANOVA) with a significance level of 5 %. ANOVA is used to determine the significance of the second-order model. The statistical significance of the second-order model is determined by F value. Whenever the calculated F value is greater than the tabulated F value, the P value will be much smaller; it indicates the significance of the statistical model. Calculated F value is defined as the mean square regression (including linear, square and interaction) and the mean square residual:

$$F\ \text{value} = \frac{\text{MS}_{\text{regression}}}{\text{MS}_{\text{residual}}} \tag{3}$$

In which:

$$\text{MS}_{\text{regression}} = \frac{\text{SS}_{\text{regression}}}{\text{DF}_{\text{regression}}} \tag{4}$$

$$\text{MS}_{\text{residual}} = \frac{\text{SS}_{\text{residual}}}{\text{DF}_{\text{residual}}} \tag{5}$$

Total degree of freedom (DF) equals the total number of experiments minus one. Regression freedom degree equals the number of sentences minus one, and the remaining freedom degree equals total degrees of freedom minus regression freedom degree.

In addition to basic analysis of variance, the program displays some additional useful information. The quantity "R-squared" is defined as:

$$R^2 = \frac{\text{SS}_{\text{Regression}}}{\text{SS}_{\text{Total}}} \tag{6}$$

We must have $0 \le R^2 \le 1$, with larger values being more desirable. There are some other R^2-like statistic displayed in the output. The "R^2-adjusted" is a variation of the ordinary R^2 statistic that reflect the number of factors in the model. A difference greater than 0.20 between the R-squared and the R^2-adjusted indicates a possible problem of the model (Montgomery 2001).

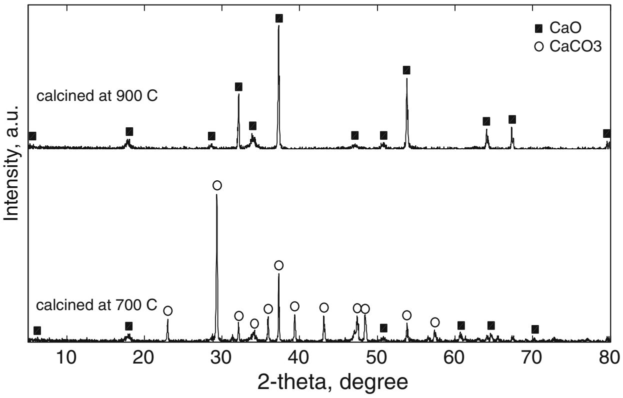

Fig. 1 XRD pattern for waste coral calcined at 700 and 900 °C for 2.5 h (*black square*) CaO; (*white circle*) $CaCO_3$

Results and discussion

Catalyst characterization

Figure 1 shows XRD patterns of the calcinated corals at 700 and 900 °C. XRD patterns showed that the catalyst calcined at 700 °C consisting $CaCO_3$, $Ca(OH)_2$ and CaO; however, the main phase was $CaCO_3$. The main phase of calcinated coral at 900 °C was CaO (Fig. 1). With increasing the calcination temperature up to 900 °C, the catalyst activity was improved, which was explained by dehydration of $Ca(OH)_2$ and transformation of $CaCO_3$ to CaO. In the similar study, Viriya-empikul et al. (2010) showed that the high temperature range above 700 °C is required to transform $CaCO_3$ to CaO. Table 3 shows the elemental chemical compositions (atomic percentage), BET surface area (S_{BET}) and total pore volume of the catalyst which it was calcined at 900 °C for 2.5 h. The value of the specific surface area and total pore volume are low. High surface area can produce internal diffusion problem due to large size of triglyceride molecules.

Statistical analysis

Regression coefficients and the constant were obtained by using the response surface methodology. Second-order

Table 3 Elemental chemical compositions (atomic percentage), BET surface area (S_{BET}) and total pore volume of the coral catalyst calcined at 900 °C for 2.5 h

Catalyst	S^{a}_{BET} (m^2g^{-1})	Total pore volume[a] (cm^3g^{-1})	Main phases[b] (wt %)	
			CaO	SrO
Coral	0.9374	0.0022	95.2	1.5

[a] Determined by N_2 adsorption analysis

[b] Determined by XRF analysis

Table 4 Standard deviation analysis (ANOVA) for model

Sources	Sum of squares (SS)	Degree of freedom (DF)	Mean of squares (MS)	*F* value	*P* value
Regression	5,439.47	9	604.385	518.34	0.000
Linear	5,012.24	3	155.101	133.02	0.000
Square	422.16	3	140.719	120.69	0.000
Interaction	5.07	3	1.689	1.45	0.334
Residual error	5.83	5	1.166		
Total	5,445.30	14			

$R^2 = 5{,}439.47/5{,}445.3 = 0.999$, R^2 (adj) = 0.997

Table 5 Assessing the significance levels of different parameters for second-order model

Factor	*P* value	Degree of significance
T	0.000	Highly significant
C	0.019	Significant
MR	0.007	Highly significant
T^2	0.000	Highly significant
C^2	0.001	Highly significant
MR^2	0.001	Highly significant
TC	0.672	Not significant
TMR	0.142	Not significant
CMR	0.341	Not significant

model was achieved using the method of least squares of error for methyl ester content (wt%) which is presented in Eq. (7):

$$Y = -798.061 + 1.831T + 5.863C + 4.096\mathrm{MR} - 0.001T^2 - 0.476C^2 - 0.099\mathrm{MR}^2 - 0.001\mathrm{TC} - 0.002\mathrm{TMR} + 0.032\mathrm{CM} \quad (7)$$

Standard deviation analysis (ANOVA) for the model is given in Table 4. As shown in this table, the total freedom degree, regression and residual error of the models are 14, 9 and 5, respectively. Comparing *F* values calculated by Eq. (3) with *F* values listed in the table, it is observed that the calculated *F* values for the model have high significant level.

R^2 and R^2-adjusted values for the model were obtained, 0.999 and 0.997, respectively. These high values are desirable and the difference between the R^2 and the R^2-adjusted considerably smaller than 0.20; therefore, these results indicate the significance of the model.

The optimum conditions from the experiments obtained were 900 °C calcination temperature, 6 wt% for the catalyst concentration and 12:1 methanol–soybean oil molar ratio (13th run at Table 2), because under these conditions, methyl ester content obtained was 100 wt%.

According to the response surface analysis and Eq. (7), the optimal values of three operating parameters were found to be 900 °C for calcination temperature, 5.6 wt% for the catalyst concentration and 12.5:1 for methanol–soybean oil molar ratio, and under these conditions,

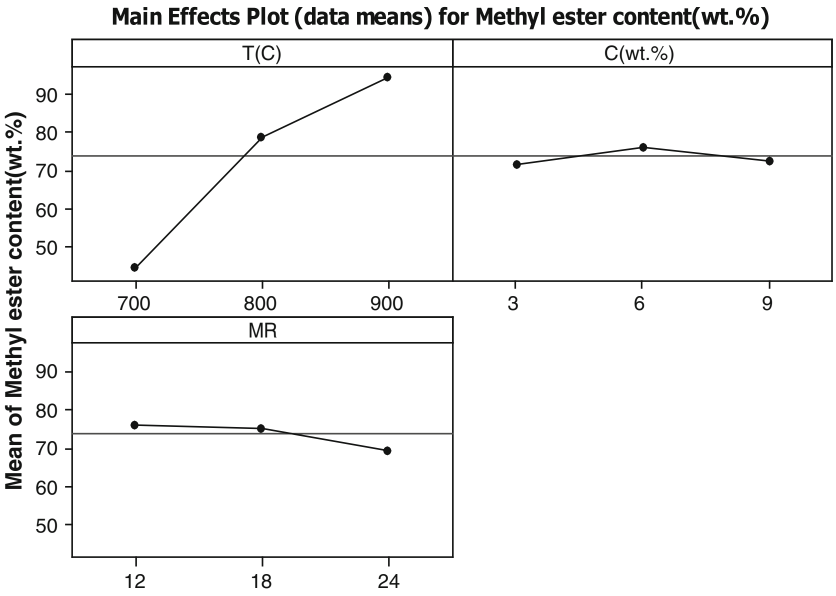

Fig. 2 Main effect plots of operating parameters on methyl ester content

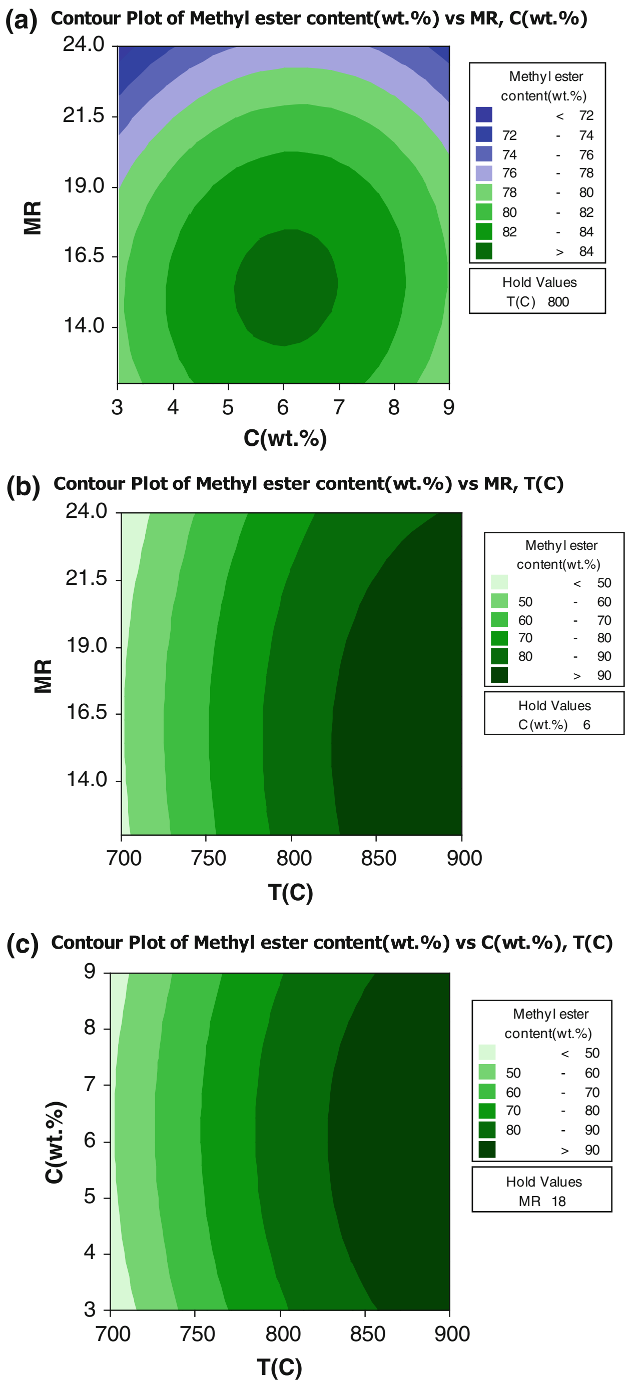

Fig. 3 Contour plots of methyl ester content (wt %):effect of operating parameters

methyl ester content obtained was 98.05 wt%. The significance of each coefficient in Eq. (7) was evaluated by the *P* value as shown in Table 5. The smaller amount of the *P* value, the more significant is the corresponding coefficient. Considering the linear effect, all three operating parameters were found to be significant terms on the methyl ester content in the final product. The calcination temperature term had the most significant linear effect, followed by the molar ratio of methanol–oil and catalyst concentration, respectively. Also, in terms of the quadratic effect, all three operating parameters were found to be significant. However, the interaction terms had no significant effect on the methyl ester content (P value > 0.05).

Effect of operating parameters

Figure 2 shows the main effects of operating parameters on methyl ester content. The calcination temperature is the most important factor, having a positive influence on methyl ester content. By increasing calcination temperature from 700 to 900 °C, calcium carbonate decomposed into calcium oxide and active basic sites were increased, especially at 900 °C.

The methanol–soybean oil molar ratio has negative influence on methyl ester content. With increasing the methanol–soybean oil molar ratio, it tends to dissolve the glycerol in methanol and separation between the methyl ester and glycerol becomes difficult. Since the transesterification is reversible in nature, this result may be due to the reverse reaction between glycerol and methyl esters produced to form the triglyceride derivatives (Ngamcharussrivichai et al. 2010). Methyl ester content is slightly increased with an increase of catalyst amount 3–6 wt%. However, with further increasing the catalyst amount above 6 wt%, the methyl ester content is slightly decreased, which might be due to saponification and mass transfer problems. As can be seen in Fig. 3, the contours are drawn at constant value of 800 °C calcination temperature, 6 wt% catalyst concentration and 18:1 molar ratio of methanol–soybean oil, respectively. From Fig. 3a, it is found that methyl ester content could be achieved higher than 84 wt% when using the molar ratio of methanol–soybean oil in the range of 13–17 and the catalyst concentration in the range of 5.1–6.9 wt%. In addition, it is found that methyl ester content increased with increasing the calcination temperature at constant value of catalyst concentration and molar ratio of methanol–oil, respectively (Fig. 3b, c).

Reusability of the catalyst

Under the optimized condition, the catalyst was reused. Before each reuse, waste catalyst was washed with methanol. The mixture of methanol and catalyst was heated at

100 °C with low stirring rate. After methanol was evaporated, catalyst was applied again. After reusing the catalyst for four times under optimal conditions, methyl ester content was obtained 100, 95.94, 82.37, 78.42 wt%, respectively. In the similar works, Wei et al. (2009) and Boey et al. (2009a) reused the waste catalyst for 13 and 11 times, respectively.

Conclusion

Using environment-friendly catalyst was particularly useful for the production of biodiesel. In this study, the waste coral was applied as a catalyst for biodiesel production. Characterization results revealed that the main component of the waste coral is calcium carbonate which transformed into calcium oxide when calcined above 700 °C. Transesterification reaction was carried out in the presence of soybean oil, methanol and waste coral in a temperature of 65 °C for 8 h. Response surface methodology was used to evaluate the effects of calcination temperature, catalyst concentration and molar ratio of methanol to oil on the methyl ester content. The optimum conditions from the experiments were obtained 900 °C for calcination temperature, 6 wt% for the catalyst concentration and 12:1 for methanol–soybean oil molar ratio. Under these conditions, methyl ester content obtained was 100 wt%. Finally, the reusability of the catalyst for 4 times was studied. After each use, the activity of waste catalyst was decreased a little. The methyl ester content reached to 78.42 wt% after the fourth reuse. It was found that waste coral shows a high potential to be used in biodiesel production process as heterogeneous base catalysts.

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