# Adsorptive removal of residual catalyst from palm biodiesel: application of response surface methodology

# Saeid Baroutian<sup>1</sup>, Kaveh Shahbaz<sup>2</sup>, Farouq Sabri Mjalli<sup>3</sup>, Inas Muen Alnashef<sup>4</sup>, Mohd Ali Hashim<sup>2</sup>

<sup>1</sup>SCION, Te Papa Tipu Innovation Park, Rotorua, New Zealand

<sup>2</sup>University of Malaya, Department of Chemical Engineering, Kuala Lumpur, Malaysia

<sup>3</sup>Sultan Qaboos University, Petroleum and Chemical Engineering Department, Muscat, Oman

<sup>4</sup>King Saud University, Department of Chemical Engineering, Riyadh, Saudi Arabia

#### Abstract

In this work, residual potassium hydroxide catalyst was removed from palm oil-based methyl esters using an adsorption technique. The produced biodiesel was initially purified through a water washing process. To produce better quality biodiesel, as well as to meet standard specifications (EN 14214 and ASTM D6751), batch adsorption on palm shell activated carbon was used for further catalyst removal. The central composite design (CCD) of the response surface methodology (RSM) was used to study the influence of adsorbent amount, time and temperature on the adsorption of potassium species. The maximum catalyst removal was achieved at 40 °C using 0.9 g activated carbon for 20 h adsorption time. The results from the Response Surface Methodology were in good agreement with the measured values. The absolute error in prediction at the optimum conditions was 3.7%, which is reasonably accurate. This study proves that adsorption post-treatment techniques can be successfully employed to improve the quality of biodiesel fuel for its effective use on diesel engines and to minimize the usage of water.

*Keywords*: biodiesel; adsorption; potassium hydroxide; optimization.

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Fatty acid methyl esters (FAME), known as biodiesel, are receiving increasing attention as an environmental friendly and promising source of energy to replace the petroleum based diesel fuel. Biodiesel is composed of monoalkyl esters of long chain fatty acids derived from renewable feedstock, such as vegetable oil or animal fats, for use in compression ignition engines [1]. A variety of vegetable oils, with different fatty acid compositions, can be used for methyl esters production. Among the vegetable oils, palm oil is a promising feedstock for biodiesel production because it tops the list of the world's vegetable oils production, and the production of biodiesel from palm oil costs lesser compared to other vegetable oils.

Currently, batchwise transesterification in stirred vessel reactors using homogeneous alkali catalysts is the most preferable process [2,3]. Basically, the purpose of the transesterification process is to lower oil viscosity through converting the large and branched fatty acids molecules into smaller and straight-chain esters molecules [4]. The most common catalysts for the transesterification reactions are the homogeneous base catalysts such as potassium and sodium hydro-

Correspondence: S. Baroutian, SCION, Te Papa Tipu Innovation Park, 49 Sala Street, Rotorua, New Zealand. E-mail: s.barout@gmail.com Paper received: 22 July, 2011 Paper accepted: 14 November, 2011 SCIENTIFIC PAPER

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xide, which are widely being used commercially in biodiesel production [5].

Although homogenous alkali transesterification is an effective method, some technical and environmental disadvantages are associated with this process. The purification steps need additional costs and generate considerable wastewater. In homogenous alkali transesterification process, for every 100 L of biodiesel produced some 20 L of wastewater is discharged [6]. This wastewater contains a low nitrogen concentration, and a high lipid and solid content. These contaminants prevent microbial growth, therefore making this wastewater problematic to degrade naturally [7].

Another aspect of the biodiesel production is the quality of this alternative fuel. Highly purified biodiesel is necessary to meet the ASTM D6751 and EN 14214 international biodiesel standards [8]. Without a doubt, a higher quality of biodiesel can reduce and minimize the engine application problems. It is proven that presence of residual alkali metals in biodiesel forms carbon deposits in fuel injection system and may poison the emission control system. In addition, it causes the blockage of the engine filter and weakens the engine in general [9]. Therefore, it is necessary to remove residual catalyst at the end of transesterification process. According to the ASTM D6751 and EN 14214 standards, the maximum allowable content of alkali metals must be less than 5 mg/kg.

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Generally, three common approaches may be adopted to purify biodiesel namely: water washing, dry washing and membrane filtration [10,11]. The most common treatment method to purify crude biodiesel is water washing, which is generally carried out to remove soap, catalyst, methanol and other contaminants from biodiesel [12]. Normally, a neutralization step neutralizing any residual catalyst and splitting the soap is carried out before the water washing action [13]. As mentioned before, the water washing technique suffers from some disadvantages, such as the increased cost and generated wastewater. Moreover, the emulsion formation can take place during processing of used cooking oils or crude feedstock containing high free fatty acids [14]. The dry washing method replaces the water with a magnesium silicate powder or an ion-exchange resin to remove the impurities. Although both methods are dry, they are applied differently [9,14]. However, using these methods, none of the products from these processes fulfills the limits specified in the EN standard. The third method for the purification of biodiesel is the membrane extraction. By using this method, the final production cost increases and throughput decreases [11,15].

Some studies have been carried out recently to study purification of biodiesel through washing. Glišić and Skala investigated different procedures of biodiesel washing using hot water [16], with the main goal to minimize the water usage in the washing-neutralization step during the biodiesel production.

Saifuddin and Chua used spray water washing at a low velocity to remove catalyst and alcohol from biodiesel [17]. Water was sprayed on top of a cylinder containing crude biodiesel and the separation process was boosted using microwave heating power (750 W).

The dry washing technique can be employed to purify crude biodiesel. This technique is usually achieved through the use of silicates (magnesol or trisyl), ion exchange resins (amberlite or purolite), cellulosics, activated clay, activated carbon, and activated fiber, and so on [18]. Adsorption on activated carbons has been investigated extensively due to their use in many applications [19,20]. Generally, activated carbons are efficient sorbents in the removal of both organic and inorganic compounds [21]. If low cost materials such as agricultural or industrial wastes are used for activated carbons preparation, they will be economical adsorbents for most of applications.

Havafuji *et al.* used activated carbon as an adsorbent to remove impurities from crude methyl esters obtained from waste vegetable oil [22]. Influences of different parameters including type of absorbent, grain size, stirring speed, temperature, etc. were investigated. In another work, Marín-Rosas *et al.* used acti-

vated carbon for desulfurization of diesel fuel with low sulphur content [23].

This study intended to reduce the production cost and to enhance the biodiesel purification process by combining the conventional water washing with a batch adsorption post-treatment. Water washing is proven to be an efficient technique to purify biodiesel. However, this method suffers from problems such as wastewater generation and high energy consumption to dry biodiesel. Therefore, it is necessary to minimize the consumption of water during biodiesel purification process. Combination of water washing and adsorption is a solution to reduce the water use in biodiesel industry and to lead a high quality biodiesel, in terms of residual alkali catalyst, to fulfill the limits specified in the EN standard.

## **EXPERIMENTAL**

#### Material

Refined palm oil was obtained locally and used as biodiesel feedstock. Methanol (99.8%) and potassium hydroxide (98.9%) were supplied by Sigma-Aldrich, Malaysia. The palm shell activated carbon was obtained from Bravo Green Sdn Bhd (Malaysia). It was produced by physical steam activation and its BET surface area, total pore volume and average pore width were 10  $m^2/g$ , 0.45 cm<sup>3</sup>/g and 2.02 nm, respectively.

#### **Biodiesel production**

The transesterification reactor included a 1500 ml jacketed glass vessel equipped with a thermometer and a reflux condenser. A digital magnetic stirrer provided the mixing requirement. A hot water circulation bath (RC6 LAUDA) was used to prepare and control the reaction temperature.

The transesterification reaction was carried out using 750 g palm oil, 7.5 g of KOH catalyst and 100% excess methanol at 60 °C and constant agitation speed of 700 rpm. After 60 min, the reaction was stopped, and the mixture was cooled and transferred to a separating funnel. The produced biodiesel was separated from the glycerol by-product by sedimentation. The excess methanol was removed by evaporation at atmospheric pressure.

#### **Biodiesel treatment**

In the pre-treatment step, biodiesel was washed with hot distilled water (60 °C). Tannic acid (0.1 wt.%) was added to water with the intention of avoid emulsion formation. The mixture of crude biodiesel and washing solution, with the volume ratio of 0.5:1 (water:biodiesel), was gently stirred. This process was continued until the ester layer became cleared. Subsequently, the mixture was transferred to a separating

funnel and the ester layer was dried using evaporation at atmospheric pressure. The crude and washed biodiesels contained 44.32 and 5.74 mg KOH/ kg, respectively.

The post-treatment of biodiesel was carried out by means of the activated carbon adsorption. Palm shell activated carbon was sieved to size range from 550 to 810  $\mu$ m, and its dirt and fines were removed by washing with deionized water. Activated carbon was then oven dried at 110 °C for 24 h, stored in desiccator, divided in separate beakers. Finally, 25 ml of washed biodiesel was added to each beaker. Activated carbon together with biodiesel was then agitated in an orbital shaker equipped with a temperature controller.

#### **Biodiesel analysis**

The content of potassium catalyst was determined before and after the adsorption post-treatment using an inductively coupled plasma optical emission spectrometer (ICP-OES Optima 7000DV, Perkin–Elmer, USA). A PerkinElmer S10 auto-sampler and a baffled cyclonic spray chamber with alumina injector were used for the analysis. The nebulizer used in this analysis was a low-flow GemCone and it was chosen based on its ability to deliver good detection limits and to handle complex samples without clogging.

The analytical instrument was calibrated using five standards (1–10 ppm) which were prepared by dissolving a potassium standard solution (500  $\mu$ g/g) in high

purity xylene. The calibration curve was linear across the entire range with a correlation coefficient of 0.99. All biodiesel samples were injected to the ICP-OES with three replications and after determining the mean value of the potassium peak area, the catalyst content in each biodiesel sample was calculated with reference to the calibration curve. The removal of catalyst (%) was calculated based on the measured initial and equilibrium concentrations.

#### **Experimental design**

In this work, Design Expert software, version 7.1 (Stat-Ease Inc., USA) was used for the experiment design and optimization of the biodiesel post-treatment. The selected design was the response surface method coupled with the central composite design. Three independent design parameters were adsorbent amount (g), time (h) and temperature (°C) and the response was the catalyst removal (%) based on the washed biodiesel catalyst content.

Because of natural variations, and in order to have a true measure of error and stable predictions, six replications of centre points in a randomized order were used. Table 1 presents the design parameters in actual and coded forms. Selection of levels for each factor was based on the preliminary studies and literature reports. The lower level of temperature was 25 °C since below that the adsorption rate is relatively slow. The upper level of temperature, 60 °C, was limited because the

Run	Туре	Adsorbent amount, g		Time, h		Temperature, °C		Determine and of
		Actual	Coded	Actual	Coded	Actual	Coded	- Potassium removal, %
19	Fact	0.01	-1	1.00	-1	25.0	-1	8.0
7	Fact	1.00	1	1.00	-1	25.0	-1	19.0
4	Fact	0.01	-1	24.00	1	25.0	-1	10.0
12	Fact	1.00	1	24.00	1	25.0	-1	22.0
20	Fact	0.01	-1	1.00	-1	60.0	1	6.0
3	Fact	1.00	1	1.00	-1	60.0	1	19.0
9	Fact	0.01	-1	24.00	1	60.0	1	9.0
18	Fact	1.00	1	24.00	1	60.0	1	20.0
14	Axial	0.26	-0.5	12.50	0	42.5	0	18.0
2	Axial	0.75	0.5	12.50	0	42.5	0	21.0
10	Axial	0.51	0	6.75	-0.5	42.5	0	16.0
16	Axial	0.51	0	18.25	0.5	42.5	0	24.0
6	Axial	0.51	0	12.50	0	33.8	-0.5	20.0
8	Axial	0.51	0	12.50	0	51.3	0.5	19.0
13	Center	0.51	0	12.50	0	42.5	0	21.0
15	Center	0.51	0	12.50	0	42.5	0	21.0
5	Center	0.51	0	12.50	0	42.5	0	20.0
17	Center	0.51	0	12.50	0	42.5	0	20.0
1	Center	0.51	0	12.50	0	42.5	0	17.0
11	Center	0.51	0	12.50	0	42.5	0	19.0

Table 1. Experimental design matrix and the response value for the biodiesel post-treatment in 25 ml adsorption system

adsorption is exothermic and the equilibrium process was not effective at higher temperature. The level of adsorption time was selected according to the adsorption equilibrium. Based on the preliminary experimentation, it was found that the adsorption equilibrium could be achieved within 15 h, for practical reasons we decided to carry out the adsorption process for 24 h. The preliminary study also indicated that removal of catalyst is too low out of the selected range adsorbent amount. Table 1 shows the conducted experiments based on the design matrix.

## **RESULTS AND DISCUSSION**

In this study, palm oil was selected as feedstock to produce biodiesel by the alkali-catalyzed transesterification. The produced biodiesel was washed with hot distillated water. The catalyst content of biodiesel was reduced from 44.32 to 5.74 mg/kg through the water washing step, which is 87% removal. Afterward, activated carbon derived from Malaysian palm shell was used as an adsorbent in a secondary step for further removal of residual potassium hydroxide catalyst from biodiesel to gain a higher quality of biodiesel. The performance of the adsorption post-treatment was evaluated in a batchwise process.

## **Response surface methodology**

The response surface methodology was used to study the single and interactive effects of parameters and to optimize the post-treatment process. This study investigates the relationship between three independent parameters (adsorbent amount, time and temperature) and the response (catalyst removal). The results from the 20-run design including catalyst removal as well as the design parameters in actual and coded forms are presented in Table 1.

Aided by response surface methodology, a model was fitted to the measured catalyst removal. The dif-

Table 2. ANOVA	for	response	surface	model
	,	response	Juliace	mouci

ference between the actual and predicted catalyst removal, indicated that no transformation is required to increase the model accuracy. The quadratic model for prediction of catalyst removal in terms of the actual factors is:

$$Removal (\%) = -6.238 + 23.132A + 0.170t + + 0.767T - 0.022At + 0.014AT - 0.0006tT - - 11.825A2 - 0.009T2 (1)$$

where A, t and T represent adsorbent amount (g), time (h) and temperature ( $^{\circ}$ C), respectively.

In order to investigate fitness and significance of the predictive model, analysis of variance (ANOVA) was performed. ANOVA presents the influence of each parameter and interaction of them on the catalyst removal. As it can be seen in Table 2, the ANOVA revealed the predictive model is very significant based on the small p-value (probability of error value), which is less than 0.0001. Furthermore, lack of fit testing produced a p-value greater than 0.01 which indicates the model explains all data well. Lack of fit is the variation of the data around the fitted model. A significant lack of fit indicates that the model is not fitting all of the design points well and a higher order model or a transformation is required. To confirm the model validity, the model prediction was compared with experimental data (Figure 1). It was found that the model was successful in capturing the correlation between the process parameters to the response with a correlation coefficient  $R^2 = 0.92$ . The adjusted  $R^2$  and adequate precision of the model were 0.86 and 12.94, respectively. The adequate precision higher than 4 confirms the adequacy of model by measuring the signal to noise ratio.

## **Effects of parameters**

The ANOVA indicates that adsorbent amount, *A*, and time, *t*, were significant to the catalyst removal due to their small p-value (less than 0.05). Although removal of the insignificant terms is tempting, they

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Source	Sum of square	Degrees of freedom	Mean of square	F-value	Prob. > <i>F</i>	Remarks
Model	449.99	8	56.25	16.08	< 0.0001	Significant
A - Adsorbent amount	276.74	1	276.74	79.09	< 0.0001	Significant
t - Time	19.88	1	19.88	5.68	0.0363	Significant
T - Temperature	3.56	1	3.56	1.02	0.3349	Not significant
At	0.13	1	0.13	0.036	0.8535	Not significant
AT	0.13	1	0.13	0.036	0.8535	Not significant
tΤ	0.13	1	0.13	0.036	0.8535	Not significant
A <sup>2</sup>	2.07	1	2.07	0.59	0.4580	Not significant
$T^2$	2.07	1	2.07	0.59	0.4580	Not significant
Residual	38.49	11	3.50	-	_	-
Lack of fit	27.87	6	4.65	2.19	0.2039	Not significant
Pure error	10.62	5	2.12	-	_	-



Figure 1. Model predicted KOH removal versus the measured response for the biodiesel post-treatment in 25 ml adsorption system.

cannot be removed because removing these terms would reduce the lack of fit p-value to 0.097 which is below the minimum desirable insignificant lack of fit of 0.1. The linear term of adsorbent amount was found to be most significant parameters due to its higher F-value.

Figures 2–4 present the three-dimensional surface plots of the predicted catalyst removal. The figures illustrate that the catalyst removal residual increases by increasing the adsorbent amount, time and temperature. However, at a higher temperature a slight reduction can be seen in the catalyst removal. The decrease in catalyst removal at higher temperatures can be attributed to the decrease in the stability of metal ionadsorbed complex. At higher temperatures metal ions desorb from activated carbon surface due to the deformation of bonds with adsorbent surface. At lower adsorbent amount and treatment time, catalyst species in the biodiesel would interact with the binding sites and thus facilitated higher adsorption. Using higher activated carbon amount and time, more catalysts are left un-adsorbed in solution due to the saturation of the binding sites.

#### Optimization

Eventually the goal of adsorption post-treatment by means of activated carbon was to maximize the removal of potassium hydroxide catalyst residual from biodiesel. For this purpose, the most desirable outcome from the predictive model was determined using nu-



Figure 2. Response surface plot of the biodiesel post-treatment in 25 ml adsorption system, catalyst removal against adsorbent amount and time.



Figure 3. Response surface plot of the biodiesel post-treatment in 25 ml adsorption system, catalyst removal against adsorbent amount and temperature.



Figure 4. Response surface plot of the biodiesel post-treatment in 25 ml adsorption system, catalyst removal against time and temperature.

merical hill-climbing algorithms [24]. All the parameters were kept in the range and the catalyst removal constraint was set to 100% for the optimization. The optimization process revealed that the maximum removal of 24.1% (based on the washed biodiesel catalyst content) can be achieved at 40  $^\circ$ C for 20 h using 0.9 g activated carbon per 25 ml of biodiesel.

To evaluate the accuracy of the predictive model, an adsorption post treatment experiment was conducted under the optimum condition and it was found that the model can accurately predict catalyst removal with an absolute error of 3.7%.

# CONCLUSION

The main objective of this study was to minimize the water consumption in the purification step during biodiesel production. Aided by adsorption post-treatment technique using palm shell activated carbon, high quality biodiesel, in terms of residual catalyst content, was produced. The response surface methodology (RSM) was used to optimize the process of adsorption post-treatment. Two parameters of adsorbent amount and time were found to be significant on the catalyst removal. The catalyst content after the washing step was reduced to 4.37 mg/kg (24% removal) using the adsorption post treatment at the optimum condition of 40 °C temperature, 0.9 g activated carbon per 25 ml of biodiesel and 20 h adsorption time. Moreover, the comparison of the optimum removal between predicted and measured values shows a reasonable accuracy with an absolute error of 3.7%. The results of this study indicated that the adsorption post-treatment is more advantageous over the conventional washing technique. The advantages can be summarised as follows:

1. Most of the washing processes use 1:1 water to biodiesel ratio while the current two-step process can significantly decrease water consumption to 50%.

2. The two-step process is able to minimize the non-degradable alkali wastewater generated during biodiesel production.

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## IZVOD

## UKLANJANJE ADSORPCIJOM ZAOSTALOG KATALIZATORA IZ BIODIZELA DOBIJENOG IZ PALMINOG ULJA: PRIMENA METODE POVRŠINE ODZIVA

Saeid Baroutian<sup>1</sup>, Kaveh Shahbaz<sup>2</sup>, Farouq Sabri Mjalli<sup>3</sup>, Inas Muen Alnashef<sup>4</sup>, Mohd Ali Hashim<sup>2</sup>

<sup>1</sup>SCION, Te Papa Tipu Innovation Park, Rotorua, New Zealand
<sup>2</sup>University of Malaya, Department of Chemical Engineering, Kuala Lumpur, Malaysia
<sup>3</sup>Sultan Qaboos University, Petroleum and Chemical Engineering Department, Muscat, Oman
<sup>4</sup>King Saud University, Department of Chemical Engineering, Riyadh, Saudi Arabia

## (Naučni rad)

U ovom radu, zaostali katalizator kalijum-hidroksid je uklonjen iz metil estara dobijenih iz palminog ulja koristeći tehniku adsorpcije. Proizvedeni biodizel je prethodno prečišćen ispiranjem sa vodom. Radi proizvodnje biodizela boljeg kvaliteta, a da bi se zadovoljili zahtevi standarda (EN 14214 i ASTM D6751), šaržna adsorpcija na aktivnom ugljeniku dobijenom iz ljuske palme je korišćena za dalje uklanjanje katalizatora. Za ispitivanje uticaja količine adsorbenta, vremena i temperature na adsorpciju kalijum-hidroksida korišćen je centralni kompozitni dizajn (CCD) metode površine odziva (RSM). Maksimalno uklanjanje katalizatora je postignuto na 40 °C za 20 h sa masom aktivnog uglja od 0,9 g. Rezultati dobijeni metodom površine odziva u dobroj su saglasnosti sa izmerenim vrednostima. Apsolutna greška predviđanja za optimalne uslovima iznosila je 3,7%, što se može uzeti kao prihvatljivo. Ova istraživanja pokazuju da se adsorpcije kao naknadni tretman može uspešno primeniti za poboljšanje kvaliteta biodizela, koji se koristi u dizel motorima, kao i za smanjenje količine korišćenja vode za ispiranje. *Ključne reči*: Biodizel • Adsorpcija • Kalijum-hidroksid • Optimizacija