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Optimisation of corncob based heterogeneous acid catalysed biodiesel synthesis using response surface methodology

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Abstract. Biodiesel, which is also known as fatty acid methyl ester (FAME) can be produced through esterification reaction of vegetable oil or animal fats catalysed by heterogeneous acid catalyst. The objective of this study was to synthesise a corncob derived carbon based heterogeneous acid catalyst functionalised by the arylation of 4-benzenediazonium sulfonate (4-BDS) for biodiesel production by using palm fatty acid distillate (PFAD) as feedstock for the esterification reaction. Subsequently, the biodiesel production reaction was optimised by using response surface methodology (RSM). RSM was employed to study the interaction between the primary factors: reaction time (2.5-6.5 h), temperature (60 to 100 °C), oil to methanol molar ratio (1: 13 to 1: 29) and catalyst loading (5 to 11 wt.%) in esterification reaction. The five-level, four factors central composite design (CCD) consisted of 30 experiments was chosen in this investigation. The predicted optimum reaction conditions was 6.48 h reaction time, 89.21 °C reaction temperature, 1 to 21.94 molar ratio of PFAD to methanol and 11 wt.% catalyst loading with 85.94% of predicted biodiesel yield. The actual optimum biodiesel yield of 83.48% was successfully achieved at the corresponding optimum operating conditions which proved the validity of the statistical optimisation model.

1. Introduction

Since 2014, world energy consumption has been increasing by more than 1% yearly due to the rapid development of world economics [1]. Following behind the industrial sector, transportation sector contributes the second highest energy consumption and it was predicted that energy demand in transportation sector will continue to grow rapidly in the next 35 years [2]. For the transportation sector, non-renewable fossil fuels is the largest energy source based on the current trend. U.S. Energy Information Administration reported that about 92.63% of the energy consumed in transportation sector originates from the petroleum derived fuels in year 2017. However, these non-renewable fossil fuels have drawbacks where combustion of fossil fuels will release large amount of greenhouse gases such as carbon dioxide into the atmosphere which will then lead to global warming.

Biodiesel, which is also known as fatty acid methyl ester (FAME), is a renewable energy that can be produced from vegetable oils or animal fats through transesterification reaction. Transesterification is also known as methanolysis where the vegetable oils or animal fats will react with alcohol in the presence of base catalyst to form FAME. However, development of biodiesel production is facing a large obstacle due to its high production cost. About 70% of the production cost is associated with the usage of high quality refined oil with low free fatty acid (FFA) content as feedstock [3]. These low



FFA content oils are required to prevent saponification reaction between the oil and base catalyst [4]. Nonetheless, usage of high FFA unrefined oil is able to significantly decrease the production cost incurred by the crude feedstock used. Biocatalyst (enzyme) such as lipase was capable to be used with oil feedstock with high FFA content without promoting saponification reaction [5]. However, enzymatic catalysts are usually expensive while enzyme catalysed reaction requires long reaction time.

In this context, low cost, non-toxic and reusable heterogeneous acid based catalyst will be more favourable without the drawbacks. Many researchers had utilised different types of biomass to synthesis carbon-based catalysts such as bamboo [4], coffee residue [6], de-oiled waste cake [7], glucose [8], microalgae residue [9], oil palm trunk and sugar bagasse [10] and had been proven to be very promising for biodiesel production. For this study, corncob was selected as the raw material as they are produced in abundance and easily available in Malaysia at all time [11]. Although oil palm waste is a more popular commercial plantation in Malaysia, researches based on this waste has been relatively saturated. To the best knowledge of the authors, very few researches on biodiesel synthesis had conducted by using corncob waste as the carbon catalyst precursor. Therefore, the feasibility of corncob to synthesis the heterogeneous catalyst for biodiesel production was being investigated in this study.

RSM which comprises of mathematical and statistical methods can be used to optimise the response of the study. Through RSM, the optimum operating conditions can be predicted based on the mathematical models at reduced number of experimental runs than the full factorial design required to carried out and in return, reducing the cost and time consumed [12]. Alejandro et al. had investigated the effect of catalyst mass, methanol to oil molar ratio and temperature on the FAME yield by using three factors central composite design (CCD) and obtained an optimised yield of 99.15% [13]. The effect of catalyst loading (sodium hydroxide and sulfuric acid) and methanol to oil molar ratio on FAME yield was performed by Anand et al. using CCD and found that methanol to oil molar ratio has a significant effect on the FAME yield [14].

A four factors five levels CCD was used in the RSM carried out in the present work to study the combination effects of operating conditions (reaction temperature, time, catalyst loading and methanol to oil molar ratio) to the FAME yield obtained through esterification reaction. CCD is a surface response design that consist of full factorial design with lesser number of runs. It requires five levels design with additional star points as compared to three levels design to provide rotatability that ensure the variance would stay constant at all points equidistance from the centre [13]. In this study, esterification reaction catalysed by the corncob derived carbon based heterogeneous acid catalyst sulfonated through arylation of 4-benzenediazonium sulfonate (4-BDS) was investigated to obtain the maximum FAME yield and the optimum operating conditions based on the mathematical model from RSM.

2. Experimental

2.1. Materials

Corn cob was obtained from the local market in Selangor, Malaysia. Palm fatty acid distillate (PFAD) was obtained from local edible oil production company. Sulfanilic acid, hydrochloric acid 37%, phosphoric acid 85%, ethanol 95%, methanol ($\geq 99.9\%$ purity) and n-hexane ($\geq 96\%$ purity) were obtained from Merck. Sodium nitrite (98.5% purity) was obtained from Acros Organics. Internal standard (methyl heptadecanoate, 99%) and external standards (methyl palmitate, 99% methyl stearate, 99% methyl oleate and 99% methyl linoleate) were all obtained from Fluka Chemie, Germany for gas chromatography (GC) quantification.

2.2. Preparation of activated carbon

Collected corncob was washed, cut into smaller pieces and dry overnight in the oven at 80 °C. Corn cob powder after ground was then impregnated with 30 v/v% phosphoric acid as the activating agent at the ratio of 1: 6 corncob to 30 v/v% phosphoric acid weight ratio for 24 h. Impregnated corncob was later subjected to thermal treatment by carbonisation in Carbolite furnace at 900 °C for 2 h at 5 °C /min. The activated carbon synthesised was denoted as CCAC900.

2.3. Synthesis of catalyst

Corncob derived carbon acid catalyst was synthesised through sulfonation method by arylation reaction of 4-BDS with activated carbon. In this process, 4-BDS will be first synthesised and directly followed by sulfonation of activated carbon with 4-BDS. Firstly, sulfanilic acid was dispersed into 300 mL of 1M HCl in a 500 mL round bottom flask in an ice bath and stirred continuously, followed by the dropwise addition of 1M sodium nitrite. The mixture was then stirred for 1 h at temperature 3 – 5 °C. Next, the solution was filtered by using vacuum pump to collect the precipitate formed, which was the 4-BDS. 4-BDS was then added into a mixture of 60 mL ethanol and 200 mL of deionised water at 3 – 5 °C. It was then followed by addition of CCAC900 and 100 mL 30 v/v% phosphoric acid and stirred for 30 min. CCAC900 will then be functionalised to form catalyst. The catalyst synthesised was denoted as Cat900.

2.4. Esterification Reaction

Esterification reaction was carried out by using PFAD and methanol as the reactant and Cat900 as the catalyst for the biodiesel production. In this reaction, 10.0 g of PFAD was kept constant and used as the basis to calculate the methanol to oil (PFAD) ratio. When the reaction is completed, the catalyst was filtered out by using filter paper and the methanol was evaporated and the remaining product (biodiesel and unreacted PFAD) was kept for analysis.

2.5. Catalyst characterisation and product analysis

Surface morphology CCAC900 and Cat900 was studied by using field emission scanning electron microscope (FESEM) JEOL, JSM-6710F at different magnifications. The functional group attached on the sample was studied by using FT-IR (Perkin Elmer Spectrum RX)-1 with KBr pellet technique at wavelength range of 400 to 4000 cm⁻¹. Biodiesel yield was investigated by using gas chromatography (GC) Perkin Elmer Claurus 500 equipped with FID detector with Nukol™ capillary column at helium gas flow rate 3 ml/min, injector temperature 250 °C of and detector temperature 220 °C. The initial oven temperature was set at 110 °C then increased to 220 °C at 10 °C/min. Biodiesel yield was calculated following equation (1) below:

$$Y = \frac{\sum C_{ME} \times m_{product}}{m_{PFAD}} \times 100\% \quad (1)$$

In equation (1), Y is the FAME yield (%), C_{ME} is the total concentration of methyl esters ($\frac{\text{g}}{\text{g product}}$), $m_{product}$ is the product mass (g product) and m_{PFAD} is the PFAD feed mass (g).

2.6. Design of Experiment

Esterification reaction in the presence of the corn cob derived acid catalyst to produce biodiesel was conducted according to the factorial design by using the response surface methodology (RSM) with Design Expert 7.0.0. A four factors and five levels central composite design (CCD) was employed in the design of the experiment. In this study, temperature (40 – 120 °C), time (0.5 – 8.5 h), catalyst loading (2 – 14 wt.%) and methanol to oil molar ratio (5:1 – 37:1) were the targeted independent variables to be investigated and FAME yield (%) from the esterification reaction was the dependent variable, which is the response of the experiment. Table 1 shows the variables and levels of the experimental design in this study. The data obtained in this study was fitted into the second order polynomial expression as show in equation (2).

$$R = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^4 \sum_{j=i+1}^4 \beta_{ij} X_i X_j \quad (2)$$

In equation (2), R is the response factor, X_i is the independent factor, β_0 is the intercept, β_i is the linear coefficient, β_{ii} is the quadratic coefficient and β_{ij} is the cross products coefficient.

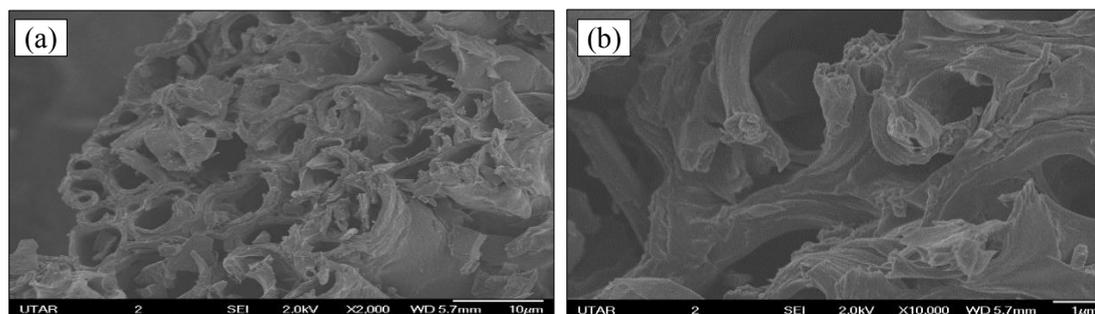
Table 1. Variables range and levels for response surface methodology.

Independent Variables	Symbols	Levels				
		$-\alpha$	-1	0	+1	$+\alpha$
Temperature ($^{\circ}\text{C}$)	A	40	60	80	100	120
Time (h)	B	0.5	2.5	4.5	6.5	8.5
Catalyst Loading (wt.%)	C	2	5	8	11	14
Methanol to oil molar ratio	D	5	13	21	29	37

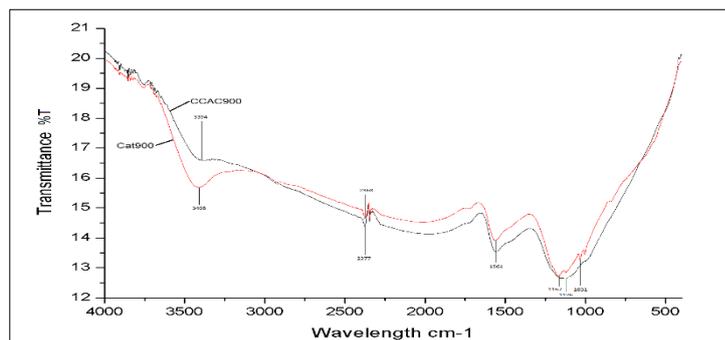
3. Results and discussion

3.1. Characterisation of catalyst

Figure 1 shows the SEM images of the surface morphology of CCAC900 at different magnification of 2000x and 10000x. As shown in the SEM image, CCAC900 had uneven and rough surface with a porous structure. The black holes observed in the SEM image represented the pores formed during activation and carbonisation [15]. The pores formed on the activated carbon is able to give good access to the reactant such as free fatty acid [6].

**Figure 1.** SEM images of CCAC900 at magnification of (a) 2000x and (b) 10000x.

The presence of active site $-\text{SO}_3\text{H}$ was confirmed by performing the FT-IR analysis. Figure 2 shows the FT-IR spectra for CCAC900 (black curve) and Cat900 (red curve) from wavelength of 400 to 4000 cm^{-1} . The FT-IR spectra for CCAC900 had a broad peak at wavelength of 1126 cm^{-1} is associated with $\text{C}=\text{O}$ stretching in acids, alcohols and phenols [7, 16]. Peak appeared at wavelength 2377 cm^{-1} may be due to the adsorption of carbon dioxide from the atmosphere on the surface of activated carbon [6]. On the other hand, the red curve in figure 2 shows the FT-IR spectra of the sulfonated activated carbon, Cat900. Overall, Cat900 exhibited similar spectra with the CCAC900. The main difference of the spectra between Cat900 and CCAC900 is that peak was observed at wavelength 1167 and 1031 cm^{-1} in Cat900 indicating asymmetric $-\text{SO}_3$ stretching and $\text{O}=\text{S}=\text{O}$ symmetric stretching modes, respectively and this is in accordance to the results reported by several literatures [4, 17, 18]. The presence of the $-\text{SO}_3$ and $\text{O}=\text{S}=\text{O}$ stretching modes confirmed the successful attachment of sulfonic group, $-\text{SO}_3\text{H}$ onto the carbon framework as the active site for esterification reaction.

**Figure 2.** FT-IR spectra of activated carbon CCAC900 and catalyst Cat900.

3.2. Quadratic Regression Model

The response (biodiesel yield) was calculated from the quadratic regression equation as shown in equation (3).

$$Y = 76.41 + 4.03A + 5.69B + 3.59 + 1.94D - 0.52AB - 0.20AC - 1.44AD - 1.18BC - 0.51BD - 0.44CD - 3.41A^2 - 2.13B^2 + 1.01C^2 - 1.43D^2 \quad (3)$$

In equation (3), Y is the biodiesel yield, A, B, C and D represent the temperature ($^{\circ}\text{C}$), time (h), catalyst loading (wt.%) and methanol to oil molar ratio, respectively. According to the regression, all four factors A, B, C and D exhibited positive effect towards the biodiesel yield where increasing the temperature, time, catalyst loading and methanol to oil molar ratio will increase the biodiesel yield in tandem. According to the results obtained from ANOVA shown in table 2, the F value of the regression model is obtained at 13.43 and the small prob > F value of less than 0.0001 and this had indicated that the model is highly significant at 95% confidence level. The terms of A (temperature), B (time), C (catalyst loading), D (methanol to oil molar ratio), A^2 , B^2 and D^2 had significant effect on the biodiesel yield. It was noted that the R-squared (R^2) of the model is 0.9260, indicating 92.6% of the variation can be explained by the model.

Table 2. Results for analysis of variance of quadratic regression

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob > F
Model	2135.68	14	152.55	13.43	< 0.0001
A: Temperature	389.49	1	389.49	34.28	< 0.0001
B: Time	776.33	1	776.33	68.32	< 0.0001
C: Catalyst Loading	309.76	1	309.76	27.26	0.0001
D: Molar Ratio	90.77	1	90.77	7.99	0.0128
AB	4.40	1	4.40	0.39	0.5429
AC	0.61	1	0.61	0.05	0.8199
AD	33.35	1	33.35	2.93	0.1073
BC	22.11	1	22.11	1.95	0.1833
BD	4.14	1	4.14	0.36	0.5550
CD	3.12	1	3.12	0.27	0.6079
A^2	318.51	1	318.51	28.03	< 0.0001
B^2	124.31	1	124.31	10.94	0.0048
C^2	28.24	1	28.24	2.49	0.1358
D^2	56.01	1	56.01	4.93	0.0422
Residual	170.44	15	11.36		
Lack of Fit	142.12	10	14.21	2.51	0.1608
Pure Error	28.32	5	5.66		
Mean	71.64954			R^2	0.926091

3.3. Effect of parameters

Figure 3(a) shows the response surface plot of temperature, methanol to oil molar ratio and FAME yield at constant time of 5.8 h and 9.6 wt.% catalyst loading. Its shows that the FAME yield increased with the increase of both temperature and methanol to oil molar ratio. The increment of FAME yield with temperature is more significant than methanol to oil molar ratio. Increasing temperature helps in driving the reaction equilibrium towards the product side and also to promote protonation of the acid catalyst in order for reaction to occur [4, 18]. At lower methanol to oil molar ratio, the increment of

FAME yield with the temperature was more obvious compared to higher methanol to oil molar ratio. At 13:1 molar ratio, the FAME yield increased from about 66 to 76.5% when the temperature increased from 60 to 100 °C while at methanol to oil molar ratio of 29:1, FAME yield increased with the temperature up to 90 °C. FAME yield decreased slightly at higher temperature of 100 °C. On the other hand, at lower temperature, the effect of methanol to oil molar on the FAME yield was more obvious at higher temperature. At 60 °C, the FAME yield increased with the methanol to oil ratio from 13:1 to 29:1 while at 100 °C, the FAME yield slightly increased from molar ratio of 13:1 and reached the optimum point at 21:1 before started to decrease. Several researches had also investigated optimum methanol to oil molar ratio at around 20:1 [7, 8, 10]. Increasing the methanol to oil molar ratio can drive the reversible reaction towards the product side but excess addition of methanol would lead to dilution of the reaction system and reduced the collision rate of oil molecules and the catalyst particles resulting in lower products formed [4, 10].

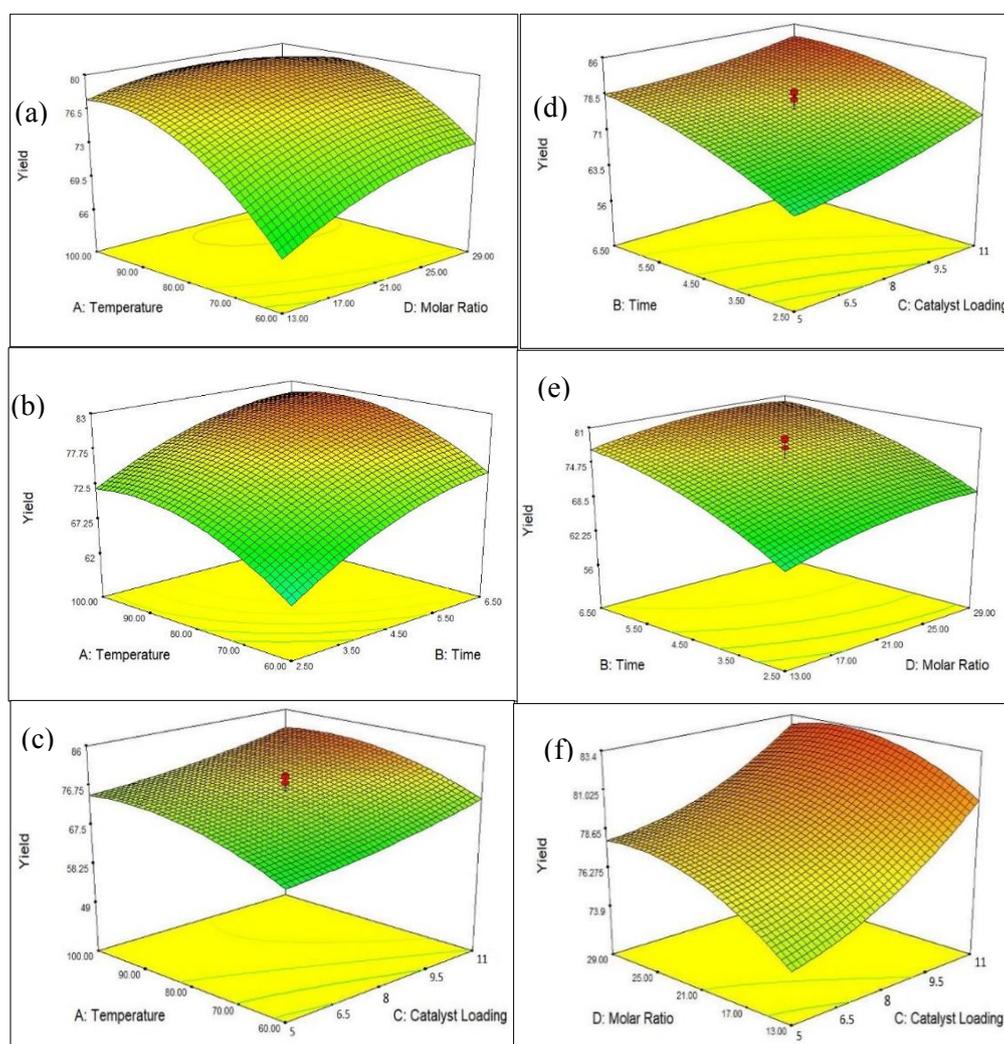


Figure 3. Response surface plot of (a) temperature (°C) and methanol to oil molar ratio (b) temperature (°C) and time (h) (c) temperature (°C) and catalyst loading (wt.%) (d) time (h) and catalyst loading (wt.%) (e) time (h) and methanol to oil molar ratio (f) catalyst loading (wt.%) and methanol to oil molar ratio on esterification reaction.

Figure 3(b) shows the response surface plot of temperature, time and FAME yield at constant methanol to oil molar ratio of 21:1 and 9.3 wt.% catalyst loading. The reaction time exhibited similar trend with the temperature whereby the FAME yield increased with reaction time. Longer reaction time is usually required to ensure a good mass transfer of catalyst in the reactants so that reaction has sufficient time to occur on the surface of catalyst [4, 19]. It shows that the FAME yield was the highest at 90 °C for all reaction time. On the other hand, it also shows that the FAME yield was always the highest at the reaction time of 6.5 h regardless of the change in temperature. In the other words, the change of temperature did not affect reaction time and vice versa. This may be due to the low interaction between these two variables. The highest FAME yield was only achieved when the temperature was at 90 °C at reaction of 6.5 h. The interaction effect of catalyst loading and temperature at time 4.5 h and 21:1 molar ratio on the FAME yield is shown in the surface response plot in figure 3(c). It shows a similar trend with figure 3(b) where the optimum temperature was at about 90 °C at all catalyst loading showing that these two are independent variables with low interaction effect [5]. The optimum temperature of 90 °C was in accordance with those reported in several literatures [4, 20].

Figure 3(d) shows the surface response plot of time, catalyst loading and FAME yield at constant temperature of 80 °C and methanol to oil molar ratio 21:1. Increment of reaction time will increase the FAME yield and the trend will not be affected by catalyst loading. On the other hand, at 2.5 h reaction time, the increment of catalyst loading will lead to the increase of FAME yield. However, when the reaction time was pro-longed to 6.5 h, increasing catalyst loading was less effective compared with increasing the FAME yield. In addition, the surface response plot of time, methanol to oil molar ratio and FAME yield at constant temperature of 80 °C and 9.3 wt.% catalyst loading was shown in figure 3(e). Overall, reaction and methanol to oil molar ratio had very less interaction as reported by Alejandro et al. [13]. It shows that the effect of methanol to oil molar ratio was more significant at short reaction time than long reaction time. At long reaction time, FAME yield only increased slightly with molar ratio. On the other hand, the effect of time on FAME yield was more obvious at lower molar ratio that the FAME yield only slightly increased with time when molar ratio was high. The effect of catalyst loading and molar ratio on the FAME yield at constant temperature of 80 °C and 5.8 h of reaction time is shown in figure 3(f). At constant temperature and time, catalyst loading had very obvious effect towards the FAME yield whereby the FAME yield increased drastically with catalyst loading. Catalyst plays an important role in lowering the activation energy of reaction, thus, increasing the amount of catalyst helps to promote the occurrence of reaction [19]. From the results obtained, methanol to oil molar ratio had lesser effect on the FAME yield. This outcome was similar with the findings reported by Dharma et al. [12]. At catalyst loading of 0.5 wt.%, the biodiesel was maximum at methanol to oil molar ratio of 26:1. However, maximum yield can be obtained at methanol to oil molar ratio of about 22:1 when the catalyst loading was increased to 11 wt.%.

3.4. Optimisation of esterification reaction

It was predicted that the maximum FAME yield of 85.94% can be achieved at the optimum condition of reaction temperature 89.24 °C, reaction time of 6.48 h, 21.94:1 methanol to oil molar ratio and 11wt.% of catalyst loading. Three experiment runs were conducted at the corresponding optimum condition and obtained an average yield of 83.48% which was close to the predicted value. The percentage error of the actual yield from the theoretical yield was only 2.86% which was considered low.

4. Conclusion

Esterification of PFAD and methanol was successfully carried out by using corncob derived carbon based heterogeneous acid catalyst. Catalyst was synthesised through sulfonation by arylation of 4-BDS with corncob derived activated carbon. RSM was carried out to study the effect of temperature, time, methanol to oil molar ratio and catalyst loading to the FAME yield of esterification reaction by using CCD. The optimum condition obtained from RSM study was at temperature 89.24 °C, reaction time of 6.48 h, 21.94:1 methanol to oil molar ratio and 11 wt.%. A highest FAME yield of 83.48%

was obtained at the optimum reaction conditions. As a whole, esterification reaction of PFAD catalysed by corncob derived heterogeneous acid catalyst has a high potential for further development in the future biodiesel industry which fulfils the sustainable framework by adding value into the waste materials.

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