optimization and validation

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Optimization And Validation Of Hydrated Magnesium Silicate On Dry Washing Purification Biodiesel Using Response Surface Methodology

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Abstract

Biodiesel is a clean energy biofuel which can be produced from waste cooking oil under transesterification and purification process by dry washing method. Dry washing purification method using hydrate magnesium silicate as a cleaning agent will improve the quality and yield of the biodiesel. This research used Response Surface Methodology with Box Behnken Design (BBD) consisting of 3 independent variables i.e. concentration of hydrate magnesium silicate, temperature and time to find the optimum condition in producing biodiesel yield. The optimum result showed that the biodiesel yield ranged between 85-94.50% where the maximum yield of 94.50% was produced under an adsorbent concentration of 2%, operating temperature of 55 ^{0}C and 20 minutes of treatment.

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Keywords: *dry washing*, optimization, yield, *response surface methodology*

1. Introduction

Biodiesel is a mono-alkyl of long chain fatty acid (triglycerides) derived from vegetable oil, animal oil or cooking oil [1],[2]. The process of producing biodiesel from cooking oil includes esterification, transesterification and purification process. Conventional purification of biodiesel as called as water washing method needs a lot of water. Under this method, washing can be carried out up to three times to remove residual impurities, glycerol residue,

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unreacted methanol and also the remaining catalyst present in biodiesel. This method has a lot of weakness, i.e. takes time, needs a lot of water and energy [3], besides that biodiesel separation using water is very difficult [4] and produces waste such as soap emulsion, glycerol, methanol and catalyst which doesn't react to environment because the washing water is directly discharged [5]. Therefore, a purification without using water 100% is needed i.e. by dry washing using cleaning agent in the form of adsorbent or resin. Dry washing purification process can overcome the weakness of water washing method. Some of them are reduce the usage of water until 100%, shorten purification process, reduce waste to environment and cheap cost. The application of this technology also affects to the lower energy usage either on purification and drying process.

Research on dry washing purification method has been conducted by Cooke *et* al. [6] and Faccini *et* al., purifying biodiesel using various adsorbent material [3], Fadhil *et* al., utilized active carbon for biodiesel purification [7], Nadambela *et* al., used ion-resin exchange for crude biodiesel purification [8], [9], used magnesium silicate in the biodiesel purification produced from transesterification [10]. Yang *et* al., used fiber bio sorbent for biodiesel purification [11]. In some previous research about crude biodiesel purification by dry washing method most of them used magnesium silicate as adsorbent. However, research on optimization on the utilization of hydrate magnesium silicate on the dry washing purification has never been conducted, whereas dry washing method was influenced by independent variables such as adsorbent concentration, temperature and time which affected biodiesel yield [12].

Concentration of adsorbent, temperature and time in the purification process must be identified as it will become the benchmark of performance and quality of the pure biodiesel which is being produced. Therefore, the most efficient statistical method for a complex optimization process i.e. Response Surface Model using Box Behnken Design was used in this research in order to find the optimum condition of independent variables (concentration of adsorbent, temperature and contact time) on the dry washing purification process [13]. Conducted optimization and modeling of Lithium Bromide-water purification on cooling process using vacuum membrane distillation (VMD) [14].

Nomenclature

Y	Yield
X_1	Concentration
X_2	Temperature
X_3	Time

2. Methodology

2.1. Tools and Material

Tools used in this research were glass ware, hot plate, magnetic stirrer, filter paper, thermometer, digital weight scale, pipette and statiff. Materials used were waste cooking oil collected from Bread Processing Unit S. I. P. Politeknik Negeri Jember, KOH P. A, methanol 100% and hydrate magnesium silicate.

2.2 Methodology

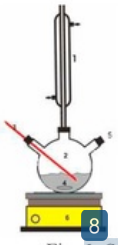
The process was started by preparing waste cooking oil of 200 ml heated at 55-60°C [15]. Then put gradually methoxide solution from mixing methanol and KOH into oil which had been heated at a pre-determined temperature by maintaining at constant temperature of 55-60 °C [16]. This transesterification reaction was carried out at methanol concentration of 60% v/v of waste cooking oil with KOH catalyst 0.5% m/v [17]. Next step was take the mixture sample of cooking oil and catalyst and then leave until reached room temperature. Separate between the biodiesel product and by-product glycerol by using separator funnel [18].

Crude biodiesel sample as transesterification product was mixed using adsorbent. Adsorbent mass amount was 1-2% m/v of biodiesel sample, the process was conducted by heating at temperature 45-65 ^{0}C for 10-20 minutes by using magnetic stirrer. Biodiesel resulted from washing process was remained until 2 hours and separated by using filter paper. Figure 1 shows a schematic diagram of the purification biodiesel with dry washing method.

2.2.1 Analytical Method

Biodiesel yield is percentage comparison between pure biodiesel (BM) as the main product with initial oil (MA) of the process. The equation of biodiesel yield according to Lani *et al.*,is as follow [19]:

$$\text{Yield (\%)} = \frac{\text{BM (mL)}}{\text{MA (mL)}} \text{x } 100\% \quad (1)$$



Description :
1. Condenser
2. Flask Chemical
3. Thermometer
4. Magnetic Stirrer
5. Insert the Adsorbent
6. Hot Plate

Fig. 1. Schematic Diagram of Biodiesel Purification with Dry Washing Method

2.2.2 Statistical analysis Method

This research used BBD (Box Behnken Design) to find the optimum condition consisted of 3 factors (independent variable) i.e. adsorbent concentration, temperature and time with the dependent variable was biodiesel yield as the response.

3. Result and Discussion

Yield produced in this research ranged between 85.00-94.50% (Table 1). In order to see the effect of independent variables (adsorbent concentration, temperature and time) to yield result, determinant test (R^2) was carried out [20], [21]. The magnitude of R^2 resulted from Minitab 17 software was 0.9783 at confidence level of 95%. R^2 = 0.9783 means that 97.83% of biodiesel yield was affected by independent variables while rest of 2.17% was affected by other factors. Mathematic model of yield as response which resulted from data processing using Minitab 17 can be seen in estimation coefficient in Figure 2. Estimation coefficient resulted second order mathematic model as follow:

$$Y = 91.167 + 0.0631X_1 + 0.750X_2 + 0.973X_3 + 1.167X_1^2 - 3.208X_2^2 + 0.667X_3^2 - 0.625X_1X_2 + 0.250X_1X_3 + 3.125X_2X_3 \quad (2)$$

Table 1. Box Behnken Design and Value of Response Yield

No.	Code			Sample Code	Independent Variables			Yield
					X_1	X_2	X_3	(%)
1	-1	-1	0	AR2	1	45	15	88.00
2	1	-1	0	CR2	2	45	15	88.50
3	-1	1	0	AT2	1	65	15	91.00
4	1	1	0	CT2	2	65	15	89.00
5	-1	0	-1	AS1	1	55	10	92.00
6	1	0	-1	CS1	2	55	10	92.50
7	-1	0	1	AS3	1	55	20	93.00
8	1	0	1	CS3	2	55	20	94.50
9	0	-1	-1	BR1	1.5	45	10	90.00
10	0	1	-1	BT1	1.5	65	10	85.00
11	0	-1	1	BR3	1.5	45	20	86.00
12	0	1	1	BT3	1.5	65	20	93.50
13	0	0	0	BS2	1.5	55	15	91.00
14	0	0	0	BS2	1.5	55	15	90.75
15	0	0	0	BS2	1.5	55	15	91.00

Coded Coefficients

Term	Effect	Coef	E Coef	T-Value	P-Value	VIF
Constant		90,917	0,363	250,29	0,000	
Concentration	0,125	0,062	0,222	0,28	0,790	1,00
Temperature	1,500	0,750	0,222	3,37	0,020	1,00
Time	1,875	0,938	0,222	4,21	0,008	1,00
Concentration*Concentration	2,583	1,292	0,327	3,94	0,011	1,01
Temperature*Temperature	-6,167	-3,083	0,327	-9,42	0,000	1,01
Time*Time	1,583	0,792	0,327	2,42	0,060	1,01
Concentration*Temperature	-1,250	-0,625	0,315	-1,99	0,104	1,00
Concentration*Time	0,500	0,250	0,315	0,79	0,463	1,00
Temperature*Time	6,250	3,125	0,315	9,93	0,000	1,00

Fig. 2. Coded Coefficients for Response Yield

Second order model for yield response in equation (1) should be tested using lack of fit test and also IIDN to prove and to accept that second order model. Testing result of lack of fit Figure 3 shows that P-value was 0.190 higher than error degree 0.05 which means that there were no lack of fit (second order model was accepted and fit).

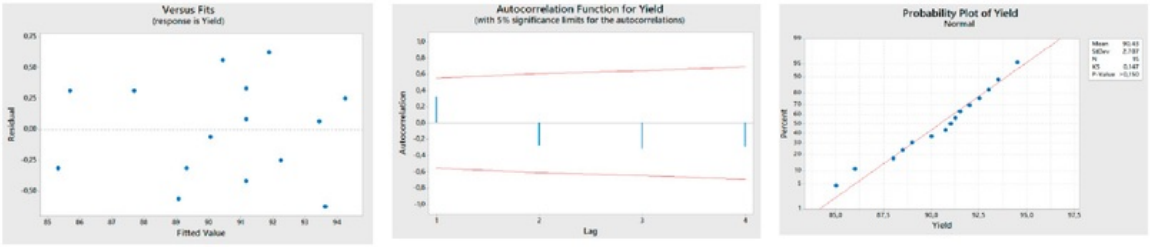
Response Surface Regression: Yield versus Concentration; Temperature; Time

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	100,329	11,1477	28,00	0,001
Linear	3	11,563	3,8542	8,64	0,020
Concentration	1	0,031	0,0313	0,07	0,802
Temperature	1	4,500	4,5000	10,09	0,025
Time	1	7,031	7,0313	15,77	0,011
Square	3	47,092	15,6639	35,01	0,001
Concentration*Concentration	1	5,026	5,0256	11,27	0,020
Temperature*Temperature	1	38,006	38,0064	85,25	0,000
Time*Time	1	1,641	1,6410	3,68	0,113
2-Way Interaction	3	40,875	13,6250	30,56	0,001
Concentration*Temperature	1	1,562	1,5625	3,50	0,120
Concentration*Time	1	0,250	0,2500	0,56	0,488
Temperature*Time	1	39,063	39,0625	87,62	0,000
Error	5	2,228	0,4458		
Lack-of-Fit	3	1,936	0,6453	4,43	0,190
Pure Error	2	0,292	0,1458		
Total	14	102,558			

Fig. 3.Analysis of Variance

Testing which done in residual assumption was IIDN (Identic, Independent, and Normal Distribution). Identic assumption could be seen from plots pattern between residual and response (fitted value) or in this case was biodiesel yield. If the resulted pattern was scatter and didn't form certain pattern then it could be said that the equation model was identic.



a b c

Fig. 4 a. Identical Residual; b. Independent Residual; c. Normal Distribution Residual

Fig. 4a shows an identic residual assumption (fit model) as it didn't form any certain pattern and relatively scatter. Fig. 4b explains Autocorrelation Function for yield. Residual assumption was called independent if the ACF value in interval $\pm\frac{2}{\sqrt{n}}$, where n value for above equation model was 15. All lag showed value lower than 0.52 and there were no lag crossing limit line.

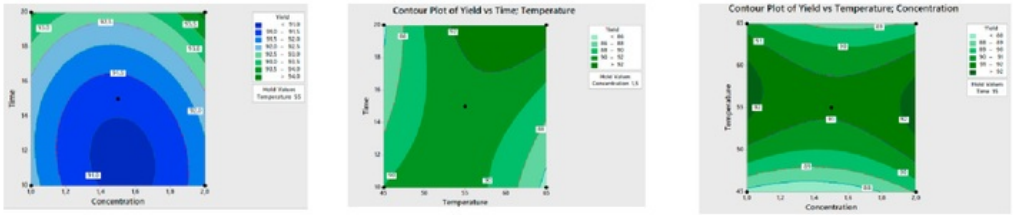
The statistical result of Kolmogorov-Smirnov (KS) in Figure 4c obtained a KS score of 0.147 less than the statistical value of n 15 i.e. 0.338 at the 0.05 confidence level so that the residual normality test has followed normal distribution. Based on the residual assumption testing of Identic, Independent and Normal Distribution which had been conducted on the equation model, it could be concluded that three residual assumption can be fulfilled or in other words the model suit.

Purification process by dry washing method was applied to biodiesel produced from transesterification by using hydrate magnesium silicate of 1%, 5% and 2%. The selection of hydrate magnesium silicate was based on the previous research [12] who found that magnesium silicate adsorbent of 1.5% could produce the highest biodiesel yield. Arifin *et al.* used adsorbent concentration 1% to produce the highest yield [9]. The selection of concentration 2% was based on the hypothesis that higher adsorbent concentration will produce higher yield, and it is proved that the highest yield of biodiesel 94.5% was produced with concentration 2% [8]. In addition, dry washing purification method is also affected by time and temperature.

Time needed for crude biodiesel purification by dry washing method is quite short if compared with wet washing method. This research used 10 minutes, 15 minutes and 20 minutes. The selection of time was based on the previous research. According to [9], 15 minutes is the most optimum time to produce the highest quality of biodiesel. The selection of 20 minutes was based on hypothesis that longer contact time used to stir crude biodiesel and adsorbent will produce the highest yield and it was proved that the highest biodiesel yield was resulted by 20 minutes treatment. This is in accordance with previous research, who produced the best biodiesel quality from stirring time

for 20-30 minutes [6].

Temperature used in this research were 45 ^{0}C, 55 ^{0}C, and 65 ^{0}C. Temperature 55 ^{0}C was found to be the most optimum temperature to produce the highest yield [12]. The highest yield in this research was 94.5% on 55 ^{0}C. Thus temperature below 55 ^{0}C will reduce yield. In temperature higher than 65 ^{0}C, hydrate magnesium silicate experiences deactivation or the performance of the adsorbent is decreasing or at temperature above 55 ^{0}C is saturated point. The contour plot of Fig. 5a visually shows the amount of biodiesel yield which produced from interaction between time and concentration. At 55 ^{0}C, the yield ranged 91-91.5%. Interaction between time and temperature in Fig. 5b, the biodiesel yield is 90-92% at 15 minutes treatment. While interaction between temperature and concentration on Fig. 5c, the biodiesel yield is 91-92% at 15 minutes treatment. Interaction between time and concentration, time and temperature, as well as temperature and concentration affected the yield result.



a b c

Fig. 5 a. Contour Plot of Time and Concentration for Yield; b. Contour Plot of Temperature and Time for Yield; c. Contour Plot of Temperature and Concentration for Yield

Yield optimization response is analytical result of temperature, time and adsorbent concentration as a whole factors affecting dry washing purification process of biodiesel. The optimization response analysis of yield is shown in Fig. 6.

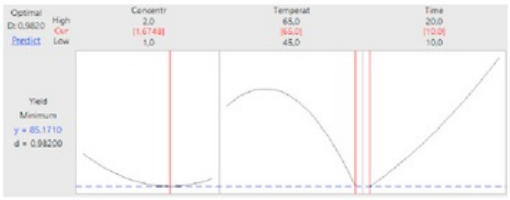


Fig. 6. Optimization Response of Temperature, Time and Concentration for Yield

Fig. 6 shows whole optimization area of biodiesel yield. The optimum condition was obtained at adsorbent concentration of 1.6748%, operating temperature 65^0 C for 10 minutes with the minimum yield y = 85.17% and composite desirability d = 0.98. The optimum result was then validated by conducting experimental test in laboratory with the result is seen on Table 2.

Table 2. The Laboratory Test of Yield from Optimization Value

No.	Operation Condition of Optimum Value			Yield Biodiesel (%)
	Concentration (%)	Temperature (^{0}C)	Times (minute)	
1	1.6748	65	10	85.00
2	1.6748	65	10	85.20
3	1.6748	65	10	85.10
4	1.6748	65	10	84.75
5	1.6748	65	10	85.00
		Average		85.01

In order to test whether the biodiesel yield resulted from laboratory test complied with the optimum value, statistic data test was conducted. Hypothesis used to test the compliance was as follow:

$H_0 : \mu_1 = \mu_0 (\mu_0 = 85.01\%)$

$H_1 : \mu_1 \neq \mu_0 (\mu_0 = 85.01\%)$

Above hypothesis was used to conduct T-test with Minitab 17 software using basic statistic tool at CI (Confidence Interval) at 95% or 0.05 and resulted T-test output as seen in Table 3.

Tabel 3. T-Test Result for Response Validation

One- Sample T: Yield								
Test of μ = 85, 01 vs ≠ 85, 01								
Variable	N	Mean	StDev	SE Mean	95%	CI	T	P
Yield	5	85.0100	0.1673	0.0748	84.8022;	85.2178	-0.00	1.000

T-test resulted in Table 3 identified that PI (Predicted Interval) from response of 84.8022% to 85.2178% with P-value was 1.00 was larger than CI (Confidence Interval) 95%, then H_0 was accepted. H_0 is accepted means that statistic found that biodiesel yield from optimization equal with the repetition test.

4. Conclusion

Based on the present research, it was concluded that biodiesel yield resulted from transesterification and purification by dry washing method could produce high biodiesel yield and the maximum optimum condition could be determined. The optimization result showed that treatment with adsorbent concentration 2%, temperature 55 ^{0}C and time 20 minutes produced the highest yield was 94.50%. While with concentration 1.68%, temperature 65 ^{0}C, and time 10 minutes produced the smallest yield was 85.17% with validation result of T-Test was 85.01%.

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