

Orthogonal experiment of biodiesel preparation from waste fatty acids

The esterification of waste fatty oils with methanol catalysed by $\text{Fe}_2(\text{SO}_4)_3$ & $\text{Zr}(\text{SO}_4)_2$ @Diatomite was planned using a four-level, five factor ($L_{16}(4^5)$) orthogonal design. Four important variables which have been identified to have larger effects on the yield of biodiesel produced from other feedstocks were investigated: MeOH to oil ratio (factor A), catalyst concentration (factor B), reaction temperature (factor C) and reaction time (factor D). The orthogonal experimental factors and levels were presented in Table S1 and the results are shown in Table S2. It can be observed that the range of the conversion varies from 61.95% to 98.56%; these data were taken as the original data and used in range analysis and univariate analysis. The range analysis data of the conversion are listed in Table S2. It can be concluded that the mass ratio of MeOH to oil has significant effect on the conversion and the optimal condition is A3B4C2D4. Therefore, the optimum conditions were obtained as follows: MeOH to oil ratio, 1.25:1; catalyst concentration, 10%; reaction temperature, 70°C; reaction time, 4 h. It reached a conversion of 98.90%. Furthermore, the univariate analysis results for the orthogonal experiment are shown Table S3. For each factor, a higher F value or R indicates that the level has a larger effect on conversion. According to the F value, the factors influencing conversion were listed in a decreasing order as follows: MeOH to oil ratio > reaction time > catalyst concentration > reaction temperature. This result was consistent with the judgment according to the R in Table S2.

Table S1. Levels and factors affecting the conversion of waste fatty oils.

Level	Factors			
	MeOH to oil ratio A (w/w)	Catalyst concentration B (respect to oil) (wt.%)	Reaction temperature C (°C)	Reaction time D (h)
1	0.5:1	4	60	1
2	1:1	6	70	2
3	1.25:1	8	80	3
4	1.5:1	10	90	4

Table S2. Results of orthogonal experiment for the optimization of biodiesel preparation from waste fatty oils.

Trial number	MeOH to oil ratio A (w/w)	Catalyst concentration B (respect to oil) (wt.%)	Reaction temperature C (°C)	Reaction time D (h)	conversion (%)
1	0.5:1	4	60	1	61.95 ± 0.24
2	0.5:1	6	70	2	78.52 ± 0.20
3	0.5:1	8	80	3	79.87 ± 0.19
4	0.5:1	10	90	4	82.08 ± 0.23
5	1:1	4	70	3	93.86 ± 0.27
6	1:1	6	60	4	95.53 ± 0.31
7	1:1	8	90	1	89.59 ± 0.17
8	1:1	10	80	2	96.00 ± 0.26
9	1.25:1	4	80	4	96.52 ± 0.24
10	1.25:1	6	90	3	97.15 ± 0.09
11	1.25:1	8	60	2	95.50 ± 0.28
12	1.25:1	10	70	1	96.05 ± 0.24
13	1.5:1	4	90	2	94.61 ± 0.31
14	1.5:1	6	80	1	91.28 ± 0.25
15	1.5:1	8	70	4	98.56 ± 0.25
16	1.5:1	10	60	3	98.45 ± 0.22
K ₁	75.61	86.74	87.86	84.72	
K ₂	93.75	90.62	91.75	91.16	
K ₃	96.31	90.88	90.92	92.33	
K ₄	95.73	93.15	90.86	93.17	
R	20.70	6.41	3.89	8.45	
A3B4C2D4					

Table S3. Univariate analysis of the orthogonal experiment

Source	Type III sum of squares	Freedom degrees	Mean square	F	Sig.
Corrected model	1470.186 ^a	12	122.515	17.058	.019
Intercept	130595.504	1	130595.504	18183.217	.000
MeOH to oil ratio	1173.174	3	391.058	54.448	.004
Catalyst concentration	84.936	3	28.312	3.942	.145
Reaction temperature	34.980	3	11.660	1.623	.350
Reaction time	177.095	3	59.032	8.219	.059
Error	21.547	3	7.182		
Total	132087.237	16			
Corrected total	1491.732	15			

a. $R^2 = .986$ (Adjusted $R^2 = .928$)