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DBSA-Catalyzed Sewage Sludge Conversion into Biodiesel in a CSTR: RSM Optimization and RTD Study

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Abstract. Sewage sludge from the primary stage of a wastewater treatment plant (WWTP) in Al-Diwaniyah City was direct-transesterified to biodiesel using dodecylbenzenesulfonic acid catalyst in a system with two continuously stirred tank reactors. The response surface and the central cube design methodologies were used to optimize the parameters affecting the biodiesel yield. The optimum conditions for the process in this study were found to be (residence time: 90 min; catalyst loading 8 wt%; methanol/oil ratio: 18; reaction temperature: 368.15 K). Under the optimum process conditions a biodiesel yield of 96.51% was obtained. The experimental residence time distribution (RTD) was calculated and compartment models were applied for the two-tank reactor system. In the CSTR, a dead zone of (10 liter) was estimated. The independent analysis of fatty acids in the sewage showed that the lipids consisted primarily of palmitic acid (C16:0 = 37.86), oleic acid (C18:1 cis-9 = 19.72) and stearic acid (C18:0 = 17.32), and the acid value was (2.44 mg KOH/g). The properties of the biodiesel produced were comparable with the ASTM D-6751-2 standard and the properties of mineral diesel.

Keywords: *CSTR; DBSA; RSM; sewage; transesterification.*

1 Introduction

The increase in the world population raises two serious issues: increased energy demand and waste accumulation. The increase in sewage sludge production (European Union) was estimated at 84% in the year 2020 compared to the production rate of 9.5 Mt in the year 2005 [1]. The sustained treatment of sludge is an emerging solution for the huge amount of disposed sewage sludge. The use of the sludge in biodiesel production also solves problems associated with the use of edible oils and algae oils [2]. The water in wastewater treatment plants consists mainly of organic and inorganic materials. In the primary treatment, the sludge is rich in non-degradable organic materials. Many researchers have investigated sewage sludge as a raw material due to its availability and cheapness. A detailed study has been conducted on different sludges from the primary and the secondary stages of wastewater treatment plants for the production of biodiesel. Moreover, three different techniques (in

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situ transesterification, extraction, extraction followed by transesterification using an acid catalyst) were investigated in order to compare between the different techniques [3]. Recently, different techniques have been used to investigate the production of biodiesel from different sewage sources [1,4-5]. Different acid and basic catalysts have been investigated but acid catalysts have been applied most extensively, especially sulfuric acid, and proved to be efficient for high-yield biodiesel production [6-16]. A Brønsted acidic ionic liquid catalyst was proved to be efficient for the conversion of sewage sludge into biodiesel [10]. The commercial production of biodiesel depends on scaling up bench-scale experiments. Most of the researchers concentrated on the investigation of the parameters affecting the biodiesel yield in batch mode. Scaling up of batch reactors cannot solve the problems associated with the continuous flow processes that are used in industry in the production of biodiesel. These processes improve the factors that affect the biodiesel production by increasing the reaction rate, mass transfer and heat transfer. Moreover, they allow continuous separation of biodiesel to be achieved [17]. In a recent review on the production of biodiesel in continuous mode, the researchers showed the advantages and disadvantages of the different types of continuous flow processes [18]. Statistical experimental design of response surface methodologies has been used in the optimization of the process parameters for biodiesel production in batch and continuous processes [19-25]. The parameters (methanol/oil ratio, residence time, and temperature) that affect the biodiesel yield were optimized in a tubular packed reactor using response surface methodology in [26]. Biodiesel production from sewage sludge in a continuous process is the right choice for converting the huge amounts of sludge being produced in wastewater treatment plants.

The present work, investigated experimentally the possibility of using sludge from the primary stage of the WWTP in Al-Diwaniyah City for biodiesel production using DBSA as catalyst in a continuously stirred tank reactor (CSTR). The design of the experiment used the central composite design and response surface methodologies for checking the effect of (residence time, catalyst loading, methanol/oil ratio, reaction temperature and mixing rate) on the biodiesel yield from sewage sludge raw material using DBSA catalyst. The design of the continuously stirred tank reactor was investigated by estimating the residence time distribution and applying compartment models. The experimental results obtained in the present work were compared with two compartment models: (1) the perfect mixing model and (2) the mixing with dead time model. The properties of the biodiesel produced were compared to the ASTM D-6751-2 standard and the properties of mineral diesel.

2 Experimental

2.1 Materials and Measuring

Chloroform (99.8), methanol (99.9%), hexane (98.8%), anhydrous $MgSO_4$ (99.8 wt %) reagents of analytical grade were procured from Sigma-Aldrech. Dodecylbenzenesulfonic acid (> 0.99) was obtained from Shanghai Hanhong Scientific Co. The standards for the quantitative analysis of biodiesel yield were procured from Sigma-Aldrech. A GC-FID (Shimadzu 2010, Japan) was used for the yield and free fatty acid analysis. The sewage sludge was obtained from the primary stage of the municipal wastewater plant of Adywaniah City and dried at 343.15 K. An independent analysis of fatty acids (%) showed that the lipids consisted primarily of capric acid (C10:0 = 3.71), lauric acid (C12:0 = 2.85), myristic acids (C14:0 = 11.06), stearic acid (C18:0 = 17.32), oleic acid (C18:1 cis-9 = 19.72), palmitic acid (C16:0 = 37.86) and others = 7.48. The specification for the instrument were (split-splitless injection of the volume = 2 μL, temperature of the oven = 483.15 K, temperature of the injector = 513.15 K, temperature of the detector = 523.15 K, ratio of splitting = 1:50, volumetric flow rate = 1 ml/min, column dimensions (film thickness = 0.25 mm, length = 30 m, inside diameter = 0.25 mm)), analysis of the water content in the sewage sludge was done in 30 min using a Karl Fischer instrument. The American Oil Chemist Society standard titration method was used for calculating the acid value of the sewage sludge oil. Experimental testing was done in triplicate and the average value was recorded.

2.2 Extraction of Oil

The sewage sludge lipids were converted to oil in a soxhlet extraction apparatus. A mixture of 600 ml of methanol and chloroform solvent was placed in a 1000 ml rounded bottom flask. The powder of the 200 g dried sewage sludge lipids was encapsulated by a filter paper and placed in the upper part of the apparatus. After being filtered, the resulted oil was extracted from the mixture using rotary evaporation and then dried at 333.15 K.

2.3 Direct Transesterification in CSTR

The experimental set-up consisted mainly of a two-tank CSTR system with specifications of (tank inside diameter: 25 cm, tank height: 50 cm), provided with a thermostat. Methanol and sewage sludge oil were introduced into the CSTR system using dosing pumps. The outlet biodiesel was collected in a product vessel. A diagram showing a schematic of the process is provided in Figure 1. A suitable amount of DBSA was added to the methanol and mixed uniformly. Then, the methanol mixture and the sewage sludge oil were heated to 333.15 K and pumped into the CSTR system. The parameters studied in the

CSTRs system were: residence time, catalyst amount, methanol to oil ratio, and reaction temperature. The yield of biodiesel was investigated at different parameter conditions and the experiments were repeated in triplicate (standard deviation less than 5% for any point). The residence time in the CSTR was calculated according to Eq. (1) [27]:

$$\tau = \frac{V_t}{v_o} = \frac{\pi.h.d^2}{4v_o} \tag{1}$$

where τ, V_t , υ_o, h, and d are residence time (min), tank volume (cm^3), flow rate (cm^3/min), height of the fluid in the reactor (cm), and inner diameter of the reactor (cm), respectively.

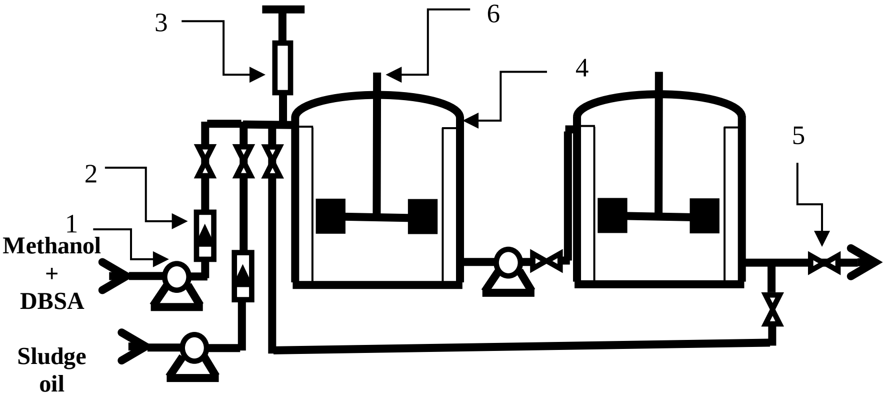

Figure 1 Schematic diagram of the CSTR system set-up: 1) dosing pump, 2) rotameter, 3) tracer syringe, 4) CSTR, 5) check valve, 6) mechanical mixer.

Afterwards, the excess alcohol was evaporated for purifying the biodiesel product using a rotary evaporator and hexane was added. Finally, the mixture was vacuum filtrated and the filtrate was dried over anhydrous $MgSO_4$. GC-FID was used for the characterization of the product obtained from the CSTR system. The tests were conducted in triplicate. The sewage sludge biodiesel yield was calculated with Eq. (2):

$$Biodiesel\,Yield(wt.\%) = \frac{W_b}{W_t} \times 100 = \frac{CF_b}{CF_s}\frac{A_b}{A_s}\frac{W_s}{W_t} \tag{2}$$

where W_b, W_t, W_s, are the mass of biodiesel, total sample, and internal standard added to the sample, respectively; A_b, A_s are the peak areas of biodiesel and the internal standard, respectively, CF_b, CF_s are the correction factors of biodiesel and the internal standard.

2.4 Process Parameter Optimization (RSM and CCD)

The range of the parameters studied was chosen according to previous studies on biodiesel production from sewage sludge raw materials [28-30]. The method of central composite design (CCD) was used to test the experimental model for lack of fit using a small number of experiments [31-32]. The response surface methodology can be summarized as consisting of three steps: 1) collection of experimental data and design of experiments that fit a quadratic model for the biodiesel yield; 2) analysis of variance and regression analysis; 3) surface and contour plots for the experimental data. A two-level, four-factor CCD was used for the optimization process in order to maximize the biodiesel yield.

RSM and the central composite design method were used for the experimental design and the optimization of the process parameters for biodiesel production using the Statistica 12 software. The parameters investigated as independent variables were: residence time (X_1), catalyst loading (X_2), methanol/oil ratio (X_3), and reaction temperature (X_4). The response function was the yield of biodiesel (Y_b). The parameters were chosen based on the most influential variables on the yield of biodiesel as stated in the literature [33]. Table 1 lists the coded values for the parameters in the present work, where the zeroes refer to center points and the +1, -1 refer to the upper and lower values.

Table 1 Parameter levels for the experimental design.

Parameter	**Symbol**	**Levels**		
		-1	**0**	**+1**
Residence time (min)	X_1	60	90	110
Catalyst loading (wt%)	X_2	4	8	12
Methanol/oil ratio (M)	X_3	15	18	21
Reaction temperature (K)	X_4	338.15	368.15	398.15

The total number of experiments that were conducted was 30 as listed in Table 2. The quadratic + 2-way option in the Statistica 12 software was chosen to solve the full quadratic model through the least residual squares method, as expressed in Eq. (3):

$$Y_b = \alpha_0 + \alpha_1 X_1 + \alpha_2 X_2 + \alpha_3 X_3 + \alpha_4 X_4 + \alpha_{11} X_1^2 + \alpha_{22} X_2^2 + \alpha_{33} X_3^2 + \alpha_{44} X_4^2 + \alpha_{12} X_1 X_2 + \alpha_{13} X_1 X_3 + \alpha_{14} X_1 X_4 + \alpha_{23} X_2 X_3 + \alpha_{24} X_2 X_4 + \alpha_{34} X_3 X_4 \qquad (3)$$

where Y_b is the yield of biodiesel, X_i are the parameters affecting the biodiesel yield, α_0, α_1, α_2, α_3, α_4 are the intercept and linear coefficients, α_{12}, α_{22}, α_{32}, α_{42} are the quadratic coefficients, and α_{12},α_{13}, α_{14}, α_{23}, α_{24}, α_{34} are the interaction coefficients.

Table 2 Experimental and predicted values of response yield with coded and uncoded parameters.

Run	Coded parameters				Uncoded parameters				Biodiesel yield, Y_b (%)		
	X_1	X_2	X_3	X_4	X_1	X_2	X_3	X_4	Experi-mental	Predicted	Deviation (%) †
1	-1	-1	-1	1	60	4	6	398.15	67.93	68.78	1.235824
2	-1	-1	1	-1	60	4	21	338.15	80.78	80.28	-0.62282
3	-1	1	-1	-1	60	12	6	338.15	89.65	89.27	-0.42567
4	-1	1	1	1	60	12	21	398.15	79.82	80.56	0.91857
5	1	-1	-1	-1	110	4	6	338.15	81.93	81.7	-0.28152
6	1	-1	1	1	110	4	21	398.15	82.26	82.76	0.604157
7	1	1	-1	1	110	12	6	398.15	76.72	77.92	1.540041
8	1	1	1	-1	110	12	21	338.15	94.84	93.52	-1.41146
9	0	0	0	0	90	8	18	368.15	96.44	96.14	-0.62409
10	0	0	0	0	90	8	18	368.15	96.52	96.14	-0.45767
11	-1	-1	-1	-1	60	4	6	338.15	82.73	82.11	-0.75508
12	-1	-1	1	1	60	4	21	398.15	71.86	72.87	1.38603
13	-1	1	-1	1	60	12	6	398.15	75.67	74.44	-1.65234
14	-1	1	1	-1	60	12	21	338.15	90.84	91.47	0.68875
15	1	-1	-1	1	110	4	6	398.15	78.91	77.75	-1.49196
16	1	-1	1	-1	110	4	21	338.15	85.54	84.79	-0.88454
17	1	1	-1	-1	110	12	6	338.15	86.63	85.39	-1.45216
18	1	1	1	1	110	12	21	398.15	89.68	88.97	-0.79802
19	0	0	0	0	90	8	18	368.15	96.51	96.14	0.592885
20	0	0	0	0	90	8	18	368.15	96.45	96.14	0.925733
21	-2	0	0	0	40	8	18	368.15	87.67	86.51	-1.34089
22	2	0	0	0	140	8	18	368.15	89.54	90.86	1.452785
23	0	-2	0	0	90	0	18	368.15	78.76	78.68	-0.10168
24	0	2	0	0	90	16	18	368.15	91.65	92.77	1.207287
25	0	0	-2	0	90	8	3	368.15	85.78	87.12	1.538108
26	0	0	2	0	90	8	33	368.15	84.83	85.08	0.293841
27	0	0	0	-2	90	8	18	308.15	72.92	73.098	0.243509
28	0	0	0	2	90	8	18	428.15	67.78	65.66	-3.22875
29	0	0	0	0	90	8	18	368.15	96.47	96.14	-0.34325
30	0	0	0	0	90	8	18	368.15	96.51	96.14	0.28084
MD											±0.959342

† Deviation (%) = (experimental-predicted)*100/experimental

‡ MD (%) $= \Sigma|deviation|/30$

2.1 Analysis of Residence Time Distribution (RTD)

Analysis of the residence time distribution was done by injecting (25 gm) of NaCl as tracer in the inlet of the CSTRs and measuring the electrical conductivity of the outlet stream as a function of time. A calibration curve was prepared for the change of conductivity with the concentration of tracer. The age function (E (t)) was calculated from the tracer concentration change with time according to Eq. (4) [34]:

$$E(t) = \frac{C(t)}{\int_0^\infty C(t)dt} \tag{4}$$

The mean time for the CSTR was calculated using Eq. (5):

$$t_m = \int_0^\infty t.E(t).dt \tag{5}$$

2.2 Analysis of Biodiesel Properties

2.2.1 Specific Gravity

The specific gravity according to the ASTM D 4052 standard was found using the hydrometer method. A graduated cylinder was filled with biodiesel and the hydrometer was left to float in the biodiesel to read the specific gravity directly.

2.2.2 Kinematic Viscosity

A Redwood viscometer was used to measure the kinematic viscosity of the biodiesel yield according to the ASTM D445 standard. First, the cup of the viscometer was filled with 50 ml of biodiesel and the orifice was closed using a valve. The viscosity was measured at a fixed temperature of 40 °C using a water bath and thermometer. The time consumed by the 50 ml of biodiesel sample to be collected from the Redwood viscometer is called the Redwood time.

2.2.3 Calorific Value

A bomb calorimeter was used according to the ASTM D2015 standard for measuring the calorific value of biodiesel sample. One gram of biodiesel sample was put in the crucible of the calorimeter and the water bath was filled with 1750 ml of distilled water. Oxygen was supplied to the system at a pressure of 25 psig for the ignition of the sample and the change of temperature over time was recorded at equal time intervals.

2.2.4 Cloud and Pour Points

The cloud and pour point was calculated according to the D 2500 standard. A jar was filled with a biodiesel sample to a height of 2.25 inches and centered with a thermometer. The jar was fixed in an ice bath at a temperature range between -1 to 2 °C. The cloud point was measured as the temperature at which the biodiesel sample starts to crystallize. Meanwhile, the pour point was recorded as the temperature at which the biodiesel sample could still be poured from the jar.

2.2.5 Flash Point

The flash point for the biodiesel was measured according to the ASTM D 93 standard. A Pensky-Marten instrument (closed cup) was used according to the D 93 standard by heating the biodiesel sample and the ignition source was directed to it at equal time intervals. The flash point temperature is the lowest temperature that provides enough ignited flammable vapor.

2.2.6 Acid Value

The acid value of the biodiesel was calculated according to the ASTM D664 standard. The test can be done by adding a suitable amount of KOH to neutralize the acids in the biodiesel sample in a titration method. The acid value is the mg of KOH required to neutralize one gram of biodiesel sample.

3 Results and Discussion

3.1 Statistical Analysis of Variance (ANOVA)

Analysis of variance was done for the empirical equation obtained by using the central composite design and the response surface methodology with confidence level higher than 95% for the response (biodiesel yield). The following equation shows the empirical model in the present study:

$$\begin{aligned} Y_b = {} & 96.14 + 3.03X_1 + 4.91X_2 + 4.63X_3 - 6.3X_4 - 1.85X_1^2 - \\ & 4.83X_2^2 - 4.67X_3^2 - 4.09X_4^2 - 0.62X_1X_2 + 2.23X_1X_3 + \\ & 1.49X_1X_4 - 0.29X_2X_3 + 0.97X_2X_4 + 2.4X_3X_4 \end{aligned} \tag{6}$$

The relation between the predicted values and experimental values is shown in Figure 2. R^2 in the figure has a value of 0.974, which indicates that the model was valid and can be used efficiently for the prediction of the biodiesel yield. Moreover, the empirical model was tested using the null hypothesis and the coefficients in the empirical equations were tested using the F-test to analyze the significance of the statistical model. The calculated F-values were compared with the tabulated F-values and were found to be greater, which validates the significance of the model, so the null hypothesis could be rejected. The rotability and orthogonality of the empirical model were calculated using an alpha (α) value of 2, where the alpha parameter is the distance to the center point and can be calculated depending on the number of parameters (n) by the formula ($2^{n/4}$) [32]. A good prediction was obtained by the model of the present study depending on the calculated and tabulated F-values (F_c = 3.8, F_t = 3.78). The model showed good fitting with a p-value of less than 0.05, which was significant at a 95%-level of confidence. The lack of fit of the model was statistically insignificant as the value of p was equal to (0.122), indicating that the model provided a satisfactory relation between the dependent and independent parameters.

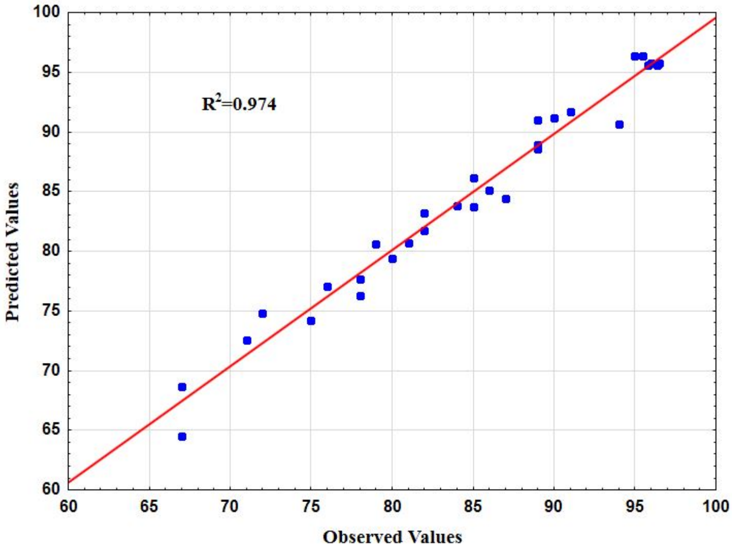

Figure 2 Predicted versus experimental values.

3.2 Variable Interaction and Surface Analysis

The interactive effect of the parameters on the biodiesel yield was investigated, as shown in Figures 3 to 8. The procedure used was holding two parameters constant and changing the other two parameters. The resulting graphs are 3D graphs called response surface graphs. Figure 3 shows the interactive influence of residence time (X_1) and catalyst loading (X_2) on the yield of biodiesel (Y_b) at constant methanol/oil ratio (X_3) and a reaction temperature (X_4) of 18 and 368.15 K, respectively. The 3D plot shows the increase of biodiesel yield with increasing residence time and catalyst loading until reaching the optimum value of 96.5%. Then, a decrease in biodiesel yield was observed for further increased residence time and catalyst loading due to the reverse transesterification reaction effect on the biodiesel yield. Mohamad, *et al.* have observed the same phenomenon [35].

Figure 4 displays the interactive influence of residence (X_1) and methanol/oil ratio (X_3) on the biodiesel yield at constant catalyst loading (X_2) and a reaction temperature (X_4) of 8 wt% and 368.15 K, respectively. Increasing the methanol/oil ratio to an excess value beyond the theoretical value of 3 would increase the yield of biodiesel due to stimulation of the forward reaction. Figure 5 proves that the interaction between the residence time (X_1) and reaction temperature (X_4) was clear at a constant catalyst loading of 8 wt% and a methanol/oil ratio of 18 and can be explained as follows. At lower residence time, the forward transesterification reaction rate increases towards the production of biodiesel until the optimum temperature of 368.15 K is reached. By increasing the residence time, the biodiesel yield is increased with increasing temperature and residence time until the optimum values of 90 min and 368.15 K are reached. The same results were reported by Miladinovi, *et al.* in [26].

The interaction between the catalyst loading (X_2) and the methanol/oil ratio (X_3) is established in Figure 6 at a constant temperature of 368.15 K and residence time of 90 min. The joint increase of the catalyst loading and the methanol/oil ratio will increase the biodiesel yield up to the optimum values of 8 wt % of catalyst loading and an 18 molar ratio of methanol/oil. Any further increase in the methanol/oil ratio will dilute the catalyst and reduce the biodiesel yield, as can be seen in the 3D plot. Ullah, *et al.* [36] reported the same result and conclusion. The interactive effect of methanol/oil ratio (X_3) and reaction temperature (X_4) is shown in Figure 7 at a constant catalyst loading of 8 wt% and residence time of 90 min. Figure 7 shows an increase in the biodiesel yield due to the increase in temperature and methanol/oil ratio up to the optimum values. Then, a decrease is observed in the biodiesel yield due to the loss and decrease of the concentration of methanol in the reaction mixture at higher temperature than 368.15 K. The combined influence of catalyst loading (X_2) and reaction temperature (X_4) is shown in Figure 8 at a methanol/oil ratio of 18 and a residence time of 90 min. The same trend as with the combined interaction between methanol/oil ratio and temperature can be seen here.

3.3 Parameters Optimization

The resulted optimized parameters using the Statistica 12 software were as follows: residence time (90 min), catalyst loading (8 wt%), methanol/oil ratio (18), and reaction temperature (368.15 K). The predicted biodiesel yield was 96.14% compared to the experimental value of 96.51% with a percentage error of 0.5% as per the validation done using the Statistica 12 software.

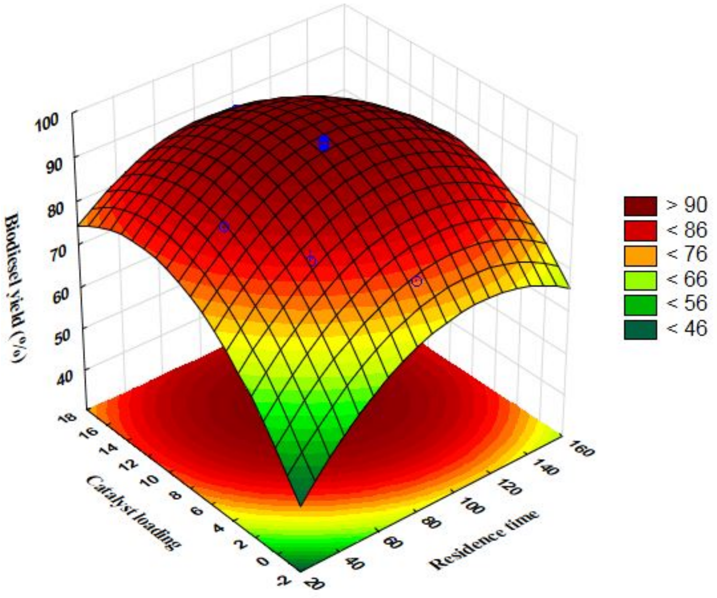

Figure 3 Influence of catalyst loading and residence time on biodiesel yield: reaction temperature 368.15 K; methanol/oil ratio 18.

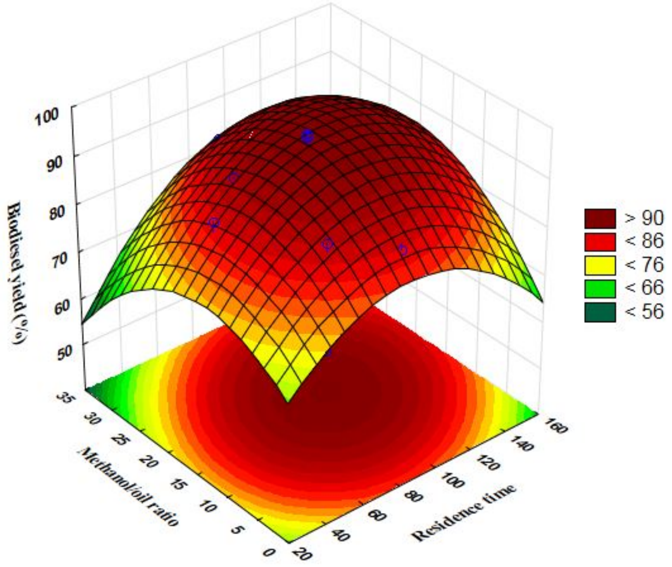

Figure 4 Influence of methanol/oil ratio and residence time on biodiesel yield: reaction temperature 368.15 K; catalyst loading 8 wt%.

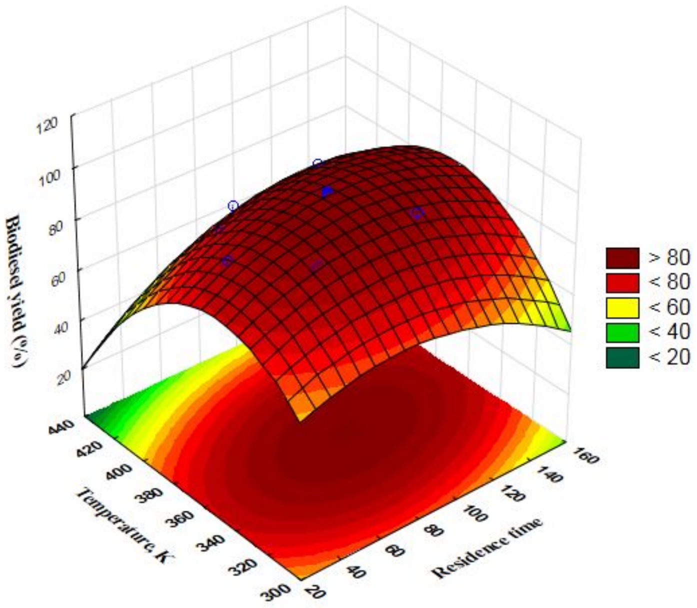

Figure 5 Influence of temperature and residence time on biodiesel yield: methanol/oil ratio 18; catalyst loading 8 wt%.

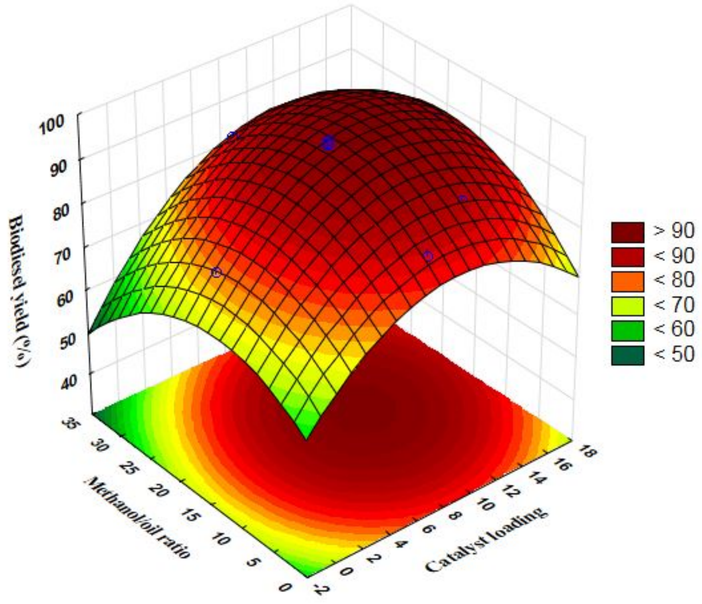

Figure 6 Influence of methanol/oil ratio and catalyst loading on biodiesel yield: residence time 90 min; reaction temperature 368.15 K.

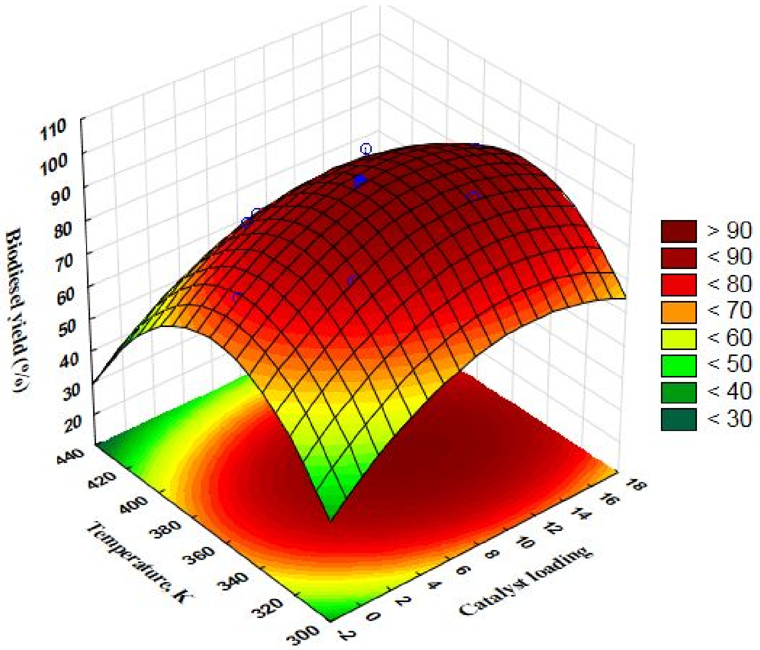

Figure 7 Influence of temperature and catalyst loading on biodiesel yield: residence time 90 min; methanol/oil ratio 18.

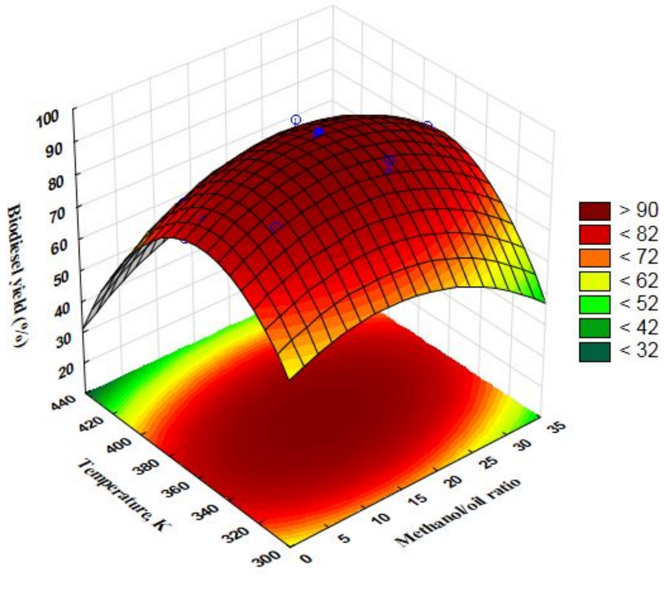

Figure 8 Influence of temperature and methanol/oil ratio on biodiesel yield: catalyst loading 8 wt %; residence time 90 min.

3.4 Analysis of Residence Time Distribution (RTD)

The experimental results obtained in the present work were compared with two compartment models: (1) the perfect mixing model [34] and (2) the mixing with dead time model [37]. The age function for the perfect mixing model was calculated with Eq. (7) [34]:

$$E_M(t) = \frac{1}{\tau_M} exp\left(-\frac{1}{\tau_M}\right) \tag{7}$$

The age function for the mixing with dead time model was obtained using Eq. (8) [37]:

$$E_{M+D}(t) = \frac{1}{\tau_M} exp\left(-\frac{(t-\tau_D)}{\tau_M}\right) \tag{8}$$

where τ_M , τ_D are the space time for the mixed and dead zones, respectively. The results for the experimental data, the perfect mixing model and the mixing with dead time model are shown in Figure 9. A lack of fit was observed for the perfect mixing model, whereas the mixing with dead time model showed a good agreement with the fitted data (R^2adjusted = 0.94). A perfectly mixed zone of (14.5 liter) was observed from the total volume of the CSTR and the other volume (10 liter) is the dead zone volume without mixing.

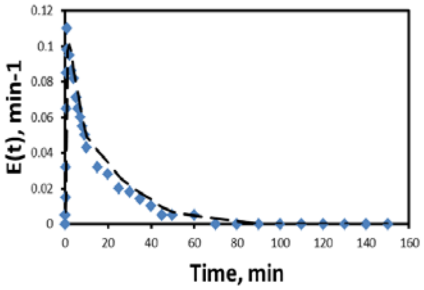

Figure 9 Age function and residence time distribution (experimental data; ----- ◆ fitted data using the mixing with dead time model).

3.5 Biodiesel Properties

The biodiesel produced from the sewage sludge was compared to the standard of biodiesel according to the ASTM D 6751-2 standard and showed the same properties as the specifications. The resulted properties of the biodiesel are listed in Table 3. The specific gravity of the biodiesel was found to be comparable to the value of diesel fuel (0.85). The kinematic viscosity is the most important property for the biodiesel yield as the reason for converting the sewage oil to biodiesel is to reduce its viscosity. The biodiesel viscosity was found to be 5.5 cSt , which is within the range recommended by ASTM D 6751-

2 (1.9-6 cSt). The calorific value of biodiesel (38.87 MJ/kg) was found to be lower than the value for diesel but within the accepted value according to ASTM D 6751-2. The calorific value of the biodiesel was reduced due to the increase of oxygen content, which reduces the carbon content. The increase of the oxygen content favors complete combustion and reduces the production of black and particulate matter. The cloud and pour point properties of biodiesel are especially important in cold places. The cloud point of the biodiesel was found to be -3 and the pour point was found to be -9. No restricted value according to ASTM D-6751-2 was recorded. The flash point is an important safety property. The flash point for the biodiesel was found to be higher than the value of standard biodiesel and mineral diesel. The acid value of the biodiesel for 0.4 mg KOH/g was found within the accepted value prescribed in the specification for standard biodiesel.

Table 3 Experimental physicochemical properties of biodiesel produced from sewage sludge.

Properties	Biodiesel	Standard ASTM D 6751-02	Diesel	Test method
Specific gravity	0.88	0.87-0.90	0.85	ASTM D4052
Viscosity at 40 °C (mm^2/s)	5.5	1.9-6.0	1.9-4.1	ASTM D445
Calorific value (MJ/kg)	38.87	–	45	ASTM D2015
Pour point (˚C)	-9	No value	-	ASTM D2500
Cloud point (˚C)	-3	No value	-12	ASTM D2500
Flash point (˚C)	170	130 °C	52	ASTM D93
Acid value (mgKOH/g)	0.4	0.8 max.	-	ASTM D664

4 Conclusion

The conversion of sewage sludge in a CSTR system was investigated using DBSA as a catalyst and gave a high yield of 96.51%. The parameters affecting the biodiesel yield were optimized using the response surface and central composite design methodologies. The model was proved to fit the experimental data with (R^2 = 0.974). The optimum conditions for the process were found to be (residence time: 90 min; catalyst loading 8 wt %; methanol/oil ratio: 18; reaction temperature: 368.15 K). Based on the conditions of the process, DBSA was found to be an efficient catalyst for the esterification and transesterification reactions in the CSTR system. For massive commercial production of DBSA it is the right choice of catalyst in a CSTR system for biodiesel production. The experimental RTD data showed a good fit with the mixing with dead time compartment model with an R^2 adjusted value = 0.94. A dead zone of (10 liter) was concluded in each CSTR of the compartment model. The physicochemical properties of the biodiesel product were in agreement with the ASTM D 6751-2 standard for biodiesel.

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