Proc. of the 5th International Conference on Power Generation Systems and Renewable Energy Technologies (PGSRET)
26-27 August, Turkey

DBSA-Catalyzed Biodiesel Production From Sewage Sludge In A Micro-Reactor: Box-Behnken Design Optimization

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***Abstract*— A specially designed microreactor device was used for the testing the process of the production of biodiesel from sewage sludge oil using the Dodecylbenzenesulfonic acid as a catalyst. The effect of co-solvent was investigated in comparison to the process of absence the co-solvent. A mixture of hexane-chloroform were used as the co-solvent in the present study. An experimental design of Box-Behnken method was used for evaluation the optimum conditions for the process. The desirability function showed that the optimum conditions for obtaining the maximum yield of biodiesel (experimental value=97.25, predicted value=97.75) were: reaction temperature (333.15 K), methanol/oil ratio (25:1), residence time (18 min), catalyst loading (7 wt%), co-solvent (16 wt %) for the microreactor system. The physicochemical properties of the sewage sludge biodiesel produced in the microreactor system showed an accepted values in accordance to the biodiesel specifications ASTM D 6751-2 and the properties of the standard diesel oil.**

Keywords—Micro-reactor, Biodiesel, Sewage sludge, Continuous transesterification, Dodecylbenzenesulfonic acid catalyst.

I. INTRODUCTION

Currently, the biodiesel production have been improved for reducing the cost of the process in the route of reducing the time and energy consumed and the other parameters effected on the process. Conventionally, the batch and continuous stirred tank reactors were used in the process for the production of biodiesel from animal fats and vegetable oils using homogeneous or heterogeneous catalyst. The researchers investigated the process of biodiesel production and showed that the main parameters effected on it where the methanol/oil ratio, catalyst type and loading, reaction temperature, residence time, type of alcohol, the stirring rate and solubility of the reactants [1-5]. A long residence time was recorded for the conventional process [6]. For solving the obstacles of high energy consumption and long residence time, the microreactor devices have been proposed as a new efficient technology in the field of microfluidic technology for biodiesel production. A great attention have been concentrated on the using the microreactors for the continuous processes including chemical reaction and this is attributed to the flexibility and swiftness of the device which provide an suitable media for the reaction to be carried out in an outstanding conditions as compared to the conventional process. The microreactors can be stacked together and used industrially using different geometries and reaction phases in catalyzed and non-catalyzed reactions [7-9]. The transesterification reaction can be done in a short time using the microreactor due to the reduction in size of the microdevice which influence the heat and mass transfer phenomena leading to the increase the rate of diffusion for the reactive materials and lower the consumption of energy [10-14]. The phenomena associated with the wall micro channel regime, surface tension, viscosity and flow rate for the multiphase flow system of the alcohol and triglycerides in the transesterification reaction [15]. The flow regimes in the microdevice was observed by Xie et al. [16] and Guan et al.[17-18] which divided into stratified flow and plug flow regimes [15]. Biodiesel production in the microreactor have been investigated at different conditions, reactive materials and catalysts and showed a promising results [10,11,19]. In the literature, a stacked microreactor or combining conventional-microreactor systems have been investigated in order to increase the biodiesel yield and reduction the residence time of the process [9,20-24]. Due to the huge amount of Sewage sludge produced daily from the municipal waste water plant. So, the Sewage sludge have been used as raw material for the production of biodiesel in many recent studies and provide a suitable biodiesel yield under a mild conditions[25-29]. Recently, biodiesel had been produced in a microchanel and enhanced by using Iso-propanol co-solvent. The reactor conditions were optimized using the Box-Behnken design (BBD) with the response surface methodology (RSM) approach [30].

The objectives of the present research is to test the micro-reactor system for the production of biodiesel from sewage sludge using dodecylbenzenesulfonic acid as a catalyst and mixture of solvents. The dodecylbenzenesulfonic acid was investigated as a catalyst for the esterification and transesterification reactions. The parameters affecting the biodiesel yield like, reaction temperature, methanol to oil ratio, residence time, catalyst amount and co-solvent amount was optimized using the box-behnken design. The reaction conditions in the micro-reactor can be applied to the stacked micro-reactors and not to the conventional reactors.

II. EXPERIMENTAL

A. Materials and Analysis

Dodecylbenzenesulfonic acid (>0.99) was procured from Shanghai Hanhong Scientific Co., Ltd. Anhydrous MgSO4 (99.8 wt. %), chloroform (99.8), methanol (99.9%), hexane (98.8%) and gas chromatography (GC) standard methyl heptadecanoate for the quantitative analysis of FAME were provided from Sigma-Aldrech. All the chemical materials used were analytical reagents. Sewage sludge was obtained from the primary stage of the municipal waste water plant of Adywaniah, Al Qadisiyah, Iraq. The sewage sludge was dried at 378.15 K. The lipid content in the sewage sludge was found 28% and the FFA content of 72%, approximately. The analysis of fatty acids (%) shows that the lipid consists 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 analysis of biodiesel produced at the optimum conditions for the free fatty acid profile and quantification are done using GC-2010-flame ionization detector (Shimadzu, Japan). Water content in Sewage sludge sample was tested with the ISO-589-1981 standard. The acid value (mg KOH/g) of the oil extracted from sewage sludge was measured using the standard titration method according with the approved method of American Oil Chemists Society (AOCS). Each test was conducted in triplicate.

B. Extraction of Oil

Extraction of oil was done in a 1000-mL round–bottom flask by mixing 200 g of sewage sludge with 600 ml of (50:50 of chloroform / methanol) as a solvent and conducted for 296 K under magnetic stirring of 600 RPM. The resulted mixture then was filtered, evaporated and finally dried at 333 K according to the literature [26].

C. Transesterification and Esterification in Micro-reactor

Continuous transesterification and esterification of sewage sludge oil was conducted in a micro-reactor (internal tube diameter= 0.5 mm, tube length= 500 mm, number of tube=100 tubes). The reactor was kept under constant temperature using a water bath at nearly atmospheric pressure. The schematic diagram of the micro-reactor device are shown in figure 1. Firstly, in the case of absence of co-solvent, methanol was mixed with a suitable amount of DBSA as a catalyst. Catalyst-methanol mixture and Sewage sludge oil was charged into the system using a glass syringe pumps. Secondly, using co-solvent (hexane-chloroform mixture, 50:50), methanol was mixed with co-solvent mixture and a suitable amount of DBSA as a catalyst. Catalyst- methanol-co-solvent mixture and Sewage sludge oil was charged into the system using a glass syringe pumps. The parameters had been studied in the micro-reactor were as follows: reaction temperature (303.15-363.15 K), methanol to oil ratio (5-45), residence time (8-28 min), catalyst amount (1-13 wt%), and co-solvent amount (0-32 wt%). The different parameter was investigated and optimized using the box-behnken design (BBD). The experiments were repeated in triplicate at the center points (standard deviation was less than 5% for any point).

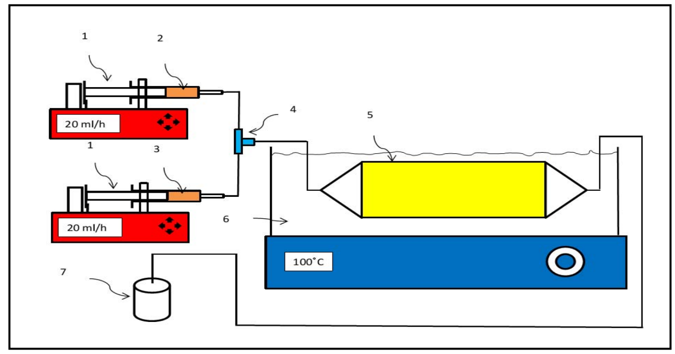

Fig. 1. Schematic diagram of the micro-reactor device set-up: 1- Syringe pump; 2- Methanol and catalyst mixture; 3- Sewage sludge oil; 4- T-shaped mixer; 5- Micro-reactor device; 6- Water bath with thermostat; 7- Biodiesel product..

Afterwards, for purifying the biodiesel product, excess alcohol was evaporated using rotary evaporator and hexane was added. Finally, The mixture was vacuum filtrated and the filtrate was dried over anhydrous MgSO4. GC-FID was used for the characterization of the product obtained from the microreactor system. The tests were conducted in triplicate. The yield of sewage sludge biodiesel was calculated according to the following equation:

$$Biodiesel\ Yield(wt.\%) = \frac{W_b}{W_t} \times 100 = \frac{CF_b}{CF_s}\frac{A_b}{A_s}\frac{W_s}{W_t} \cdots (1)$$

Where Wb, Wt, Ws, are the mass of biodiesel, total sample, and internal standard added to the sample, respectively, Ab, As is the peak area of biodiesel, and internal standard, respectively, CFb, CFs is the correction factor of biodiesel, and internal standards.

D. Process Parameter Optimization (BBD and RSM)

The parameters was examined using the response methodology along with the Box-Behnken design. A small experimental data is needed in the case of Box-Behnken design and it is considered as a suitable method for the optimization of parameters than other methods of factorial and composite designs [31]. The parameters effecting on the biodiesel yield from sewage sludge was studied in a range chooses depending on the previous literature [32-34]. The method of Box-Behnken design (BBD) is designed to test the experimental model for the lack of fit using a small number of experiments [35-36]. Response surface methodology can summarized by three steps (1.Collection of experimental data and design of experiments that fit a quadratic model for the yield of biodiesel; 2. Analysis of variance and regression analysis; 3. Surface and contour plots for the experimental data). A three-level, five-factor BBD had been used for the optimization process in order to maximize the biodiesel yield. RSM and Box-Behnken design method were used for the experimental design and the optimization of the process parameters for biodiesel production using STATISTICA 12 software. The parameters investigated as independent variables were reaction temperature (X_1), methanol/oil ratio (X_2), residence time (X_3), Catalyst loading (X_4), Co-solvent (X_5) and the response function was the yield of biodiesel (Y). the parameters effected on biodiesel yield were chosen due to be the most influenced variables on the yield of biodiesel as stated in the literature [35-40]. Table 1 list the coded values for the parameters in the present work where the zeroes refer to the center points and the +1, -1 refer to the upper and lower values. The total number of experiments had been applied were 46 experiment. The 2-way interactions (quadratic + linear) option in the STATISTICA 12 software was chosen to solve for the full quadratic model through the least residual squares method as in the equation (2):

$$Y = \alpha_0 + \alpha_1 X_1 + \alpha_2 X_2 + \alpha_3 X_3 + \alpha_4 X_4 + \alpha_5 X_5 + \alpha_{11} X_1^2 + \alpha_{22} X_2^2 + \alpha_{33} X_3^2 + \alpha_{44} X_4^2 + \alpha_{55} X_5^2 + \alpha_{12} X_1 X_2 + \alpha_{13} X_1 X_3 + \alpha_{14} X_1 X_4 + \alpha_{15} X_1 X_5 + \alpha_{23} X_2 X_3 + \alpha_{24} X_2 X_4 + \alpha_{25} X_2 X_5 + \alpha_{34} X_3 X_4 + \alpha_{35} X_3 X_5 + \alpha_{45} X_4 X_5 \cdots (2)$$

Where Y is the yield of biodiesel, X are the parameter affecting on the biodiesel yield, $\alpha_0, \alpha_1, \alpha_2, \alpha_3, \alpha_4, \alpha_5$ are the intercept and linear coefficients, $\alpha_1^2, \alpha_2^2, \alpha_3^2, \alpha_4^2, \alpha_5^2$ are the quadratic coefficients, $\alpha_{12}, \alpha_{13}, \alpha_{14}, \alpha_{15}, \alpha_{23}, \alpha_{24}, \alpha_{25}, \alpha_{34}, \alpha_{35}, \alpha_{45}$ are the interaction coefficients.

TABLE I. THE PARAMETERS LEVEL FOR THE EXPERIMENTAL DESIGN

Parameter	Symbol	Levels		
		-1	0	+1
Temperature (K)	X_1	303.15	333.15	363.15
Methanol/oil ratio	X_2	5	25	45
Time (min)	X_3	8	18	28
Catalyst (wt%)	X_4	1	7	13
Co-solvent (wt%)	X_5	0	16	32

E. Analysis of Biodiesel Properties

According to the specifications (ASTM D 4052), the specific gravity was found using the hydrometer method. A graduated cylinder was filled with biodiesel and the hydrometer left to float in the biodiesel to read the specific gravity directly. A Redwood viscometer was used to measure the kinematic viscosity for biodiesel according to the ASTM D445. A bomb calorimeter was used according to the ASTM D2015 for measuring the calorific value of biodiesel sample. The cloud and pour point was calculated according to the specification D 2500. The flash point for the biodiesel was measured according to the ASTM specifications no. D 93 using an instrument called Pensky-Marten (closed cup). The acid value for the biodiesel was calculated according to the specification ASTM D664.

III. RESULTS AND DISCUSSION

A. Statistical Analysis of Variance (ANOVA)

The analysis of variance was done for the empirical equation obtained by using the response surface methodology and Box-Behnken design with a confidence level of 95% for the response (yield of biodiesel). The following equation shows the empirical model in the present study:

$$Y = 58.73 + 0.56X_1 + 1.75X_2 - 0.69X_3 + 0.5X_4 + 5.25X_5 + 7.37X_1^2 + 5.83X_2^2 + 2.11X_3^2 + 4.91X_4^2 + 8.74X_5^2 + 2X_1X_2 - 1.5X_1X_3 + 0.5X_1X_4 + 3.25X_1X_5 - 0.5X_2X_3 + 1.5X_2X_4 + 7.5X_2X_5 + 0.25X_3X_4 + 6.5X_3X_5 + 2.25X_4X_5 \cdots (3)$$

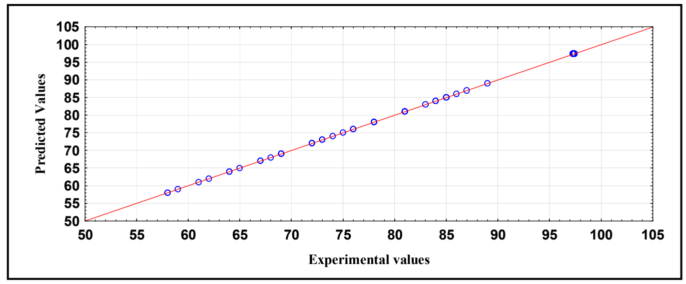

Fig. 2. Influence of alcohol/biomass ratio (v/w) on the yield of biodiesel.

The relation between the predicted values and experimental values is shown in the figure 2. The value for coefficient of determination (R^2) and adjusted coefficient (adj. R^2) in the figure showed a values of 99.8% and 99.5%, respectively, which indicate that the model validated and can be used efficiently for the prediction the yield of biodiesel. Moreover, the empirical model had been tested using the null hypothesis and the coefficients in the empirical equations was tested using the F-test to analyze the significance for the statistical model. The calculated F-values was compared with the tabulated F-values and was found greater than it which indicates the significance of the model and the null hypothesis can be rejected. A good prediction was obtained by the model of the present study depending on the values of calculated and tabulated F-values (F_c=7.5, F_t=7.28). The model shows a good fitting with a p-value of less than 0.05 which is significant at 95% level of confidence. The lack of fit for the model was insignificant statistically as the value of p was equal to (0.092) indicating that the model provide a satisfactory relation between the dependent and independent parameters. The values of pure and residual errors was found very low indicating that the experimental data is reproducible and the model educate to represent the relationship between the dependent and independent parameters.

B. Variable Interaction and Surface Analysis

The significance of the independent factors (Main and interacted effect) was determined also as shown in table 3 using the ANOVA analysis. The analysis showed that the values of p for all the main and interacted factors were less than 0.05 indicating that the factors statistically significant remembering that the smaller the values of p means the larger the statistical significance for the factors [41]. Except the terms of quadratic catalyst loading, interacted linear temperature by linear catalyst loading, interacted linear methanol/oil ratio by linear residence time, interacted linear residence time by linear catalyst loading which getting a values of p more than 0.05 so that it is not included in the model equation (Eq. 3). The interacted parameter effects in the model equation (3) was shown in the 3D figures 3-12. The sewage sludge biodiesel yield was noticeably increased as compared to the case of absence the co-solvent mixture which was done at an elementary experiment. The increase of biodiesel yield can be interpreted by the improving the solubility of sewage sludge oil in the reactant mixture (figures 6,9,11,12). Further increase in the amount of co-solvent beyond the optimum value of (16 wt%) may dilute the reactant mixture [42]. The main influence of methanol/oil ratio on the yield of sewage sludge biodiesel can be seen approximately as a linear proportional relation due to the improvement of the mass transfer area (interfacial) between the reactants which shift the reactants in the forward direction of the equilibrium reaction [43]. The influence of residence time on the yield of biodiesel was found of positive effect until the 18 min. Increasing the residence time more than the value of 18 min may increase the yield of biodiesel slightly due to the reaching the point of equilibrium [44]. A minor negative interacted effect of the co-solvent-residence time was concluded from the 3D figures 6,9,11,12 due to the phenomena of dilution [45]. Also, a negative interacted effect for the methanol/oil ratio-residence time was observed in the figure 3. At the lower level of residence time (8 min), any increase in the methanol/oil ratio showed a rapid increase in the biodiesel yield due to providing the excess amount of methanol in the reaction system which shift the reaction in the direction of formation the biodiesel. At the higher level of residence time (28 min), the increase of methanol/oil ratio beyond the value of 25:1 showed a decrease in the biodiesel yield. The increase in the methanol/oil ratio would increase the rate of mass transfer through the reactant phases [46]. The 3D figure 10 shows the interacted effect of catalyst loading-residence time on the yield of sewage sludge biodiesel. An increase in the biodiesel yield was observed due to the co-increase of catalyst loading and residence time until reaching the optimum value followed by a decrease in the biodiesel yield due to the reversing the direction of the reaction [38]. The interacted effect of residence time-reaction temperature on the biodiesel yield was shown in the 3D figure 4. A co-increasing of the temperature and residence time led to the increase of biodiesel yield until reaching the optimum value [39]. The same scenario was observed for the co effect of catalyst loading and methanol/oil ratio on the biodiesel yield (figure 8) which also observed by Ullah et al. [40]. Moreover, interacted effect of methanol/oil ratio and reaction temperature on the biodiesel yield showed an increase in biodiesel yield with increasing of temperature and methanol/oil ratio with a decrease in the biodiesel yield beyond the optimum value due to the decrease in the concentration of methanol in the reaction mixture due to the vaporization of methanol (Figure 3). Also, the influence of catalyst loading and reaction temperature on the biodiesel yield (figure 5) was observed to show the same scenario of that observed with the increasing the methanol/oil ratio and temperature.

C. Parameters Optimization

the statistical results of the experimental design used in the present study (Box-Behnken Design) showed that the optimum conditions for obtaining the maximum yield of biodiesel (experimental value=97.25, predicted value=97.75). Using the desirability function, the optimum condition values of reaction temperature (333.15 K), methanol/oil ratio (25:1), residence time (18 min), catalyst loading (7 wt%), co-solvent (16 wt %) were found for the microreactor system. For the validation of the results, an experiment was done using the optimum conditions and showed an error value of less than (1%).

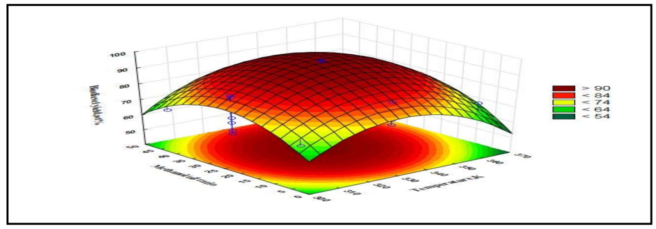

Fig. 3. Influence of methanol/oil ratio and reaction temperature on biodiesel yield.

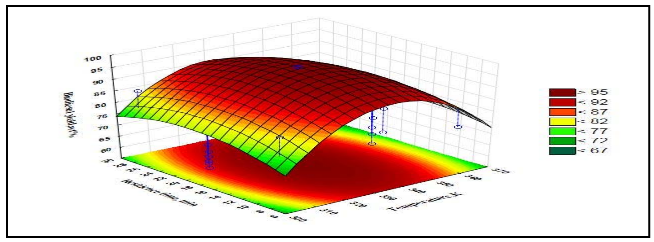

Fig. 4. Influence of reaction temperature and residence time on biodiesel yield..

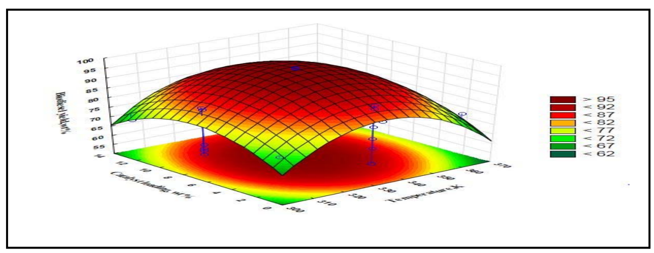

Fig. 5. Influence of reaction temperature and catalyst loading on biodiesel yield.

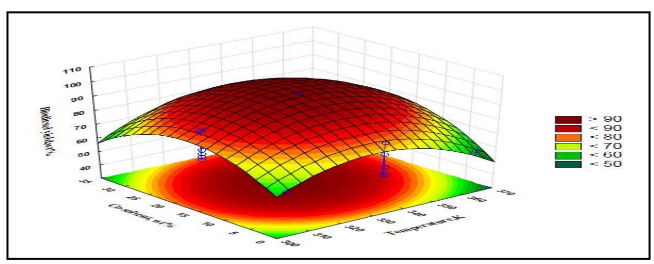

Fig. 6. Influence of co-solvent and reaction temperature on biodiesel yield.

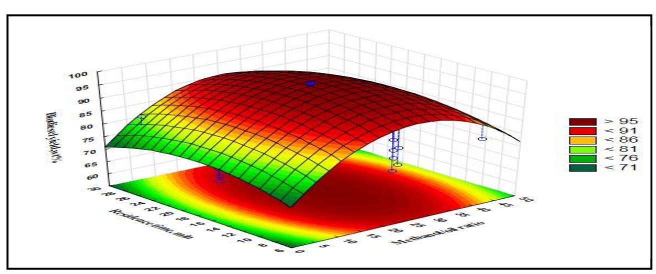

Fig. 7. Influence of residence time and methanol/oil ratio on biodiesel yield.

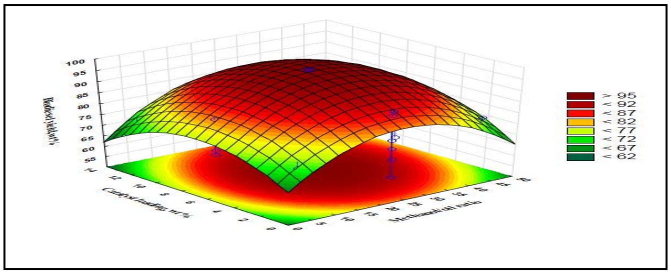

Fig. 8. Influence of catalyst loading and methanol/oil ratio on biodiesel yield

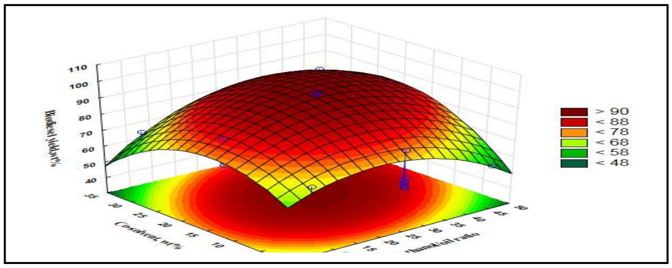

Fig. 9. Influence of co-solvent and methanol/oil ratio on biodiesel yield

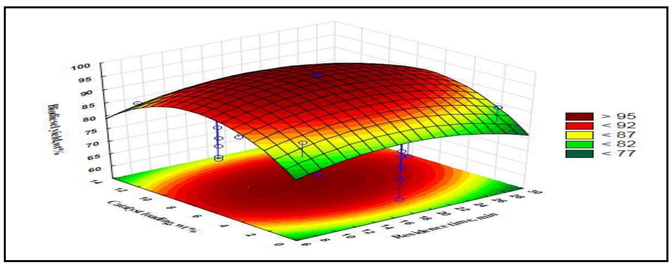

Fig. 10. Influence of catalyst loading and residence time on biodiesel yield

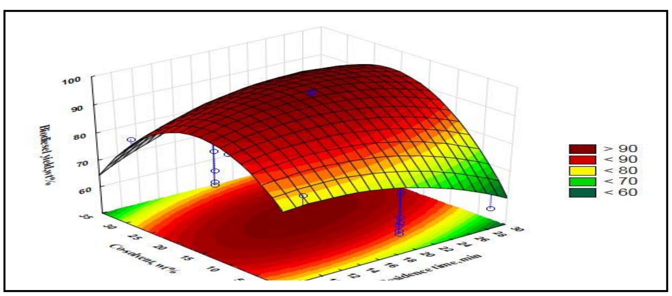

Fig. 11. Influence of co-solvent and residence time on biodiesel yield

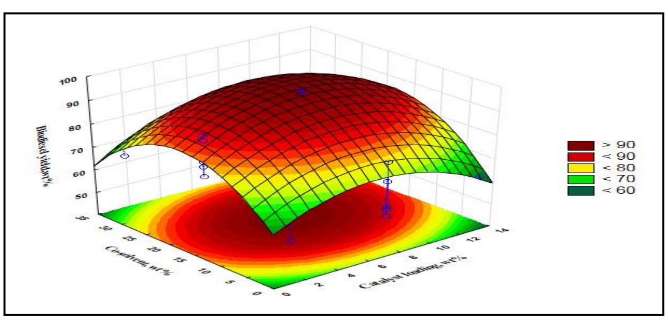

Fig. 12. Influence of co-solvent and catalyst loading on biodiesel yield

D. Biodiesel Properties

The properties of the biodiesel produced from sewage sludge was compared to the standard properties of biodiesel according to the specifications ASTM D 6751-2 and showed an accepted properties according to the specifications. The resulted properties of biodiesel were listed in the table 2. The specific gravity of biodiesel was found in 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 in order to reduce the viscosity. The biodiesel viscosity was found to be 5.4 cSt and it is within the range recommended by the ASTM D 6751-2 (1.9-6 cSt). The calorific value of biodiesel (37.85 MJ/kg) was found less than the value for the diesel but in the accepted value according to the ASTM D 6751-2. The calorific value of biodiesel is reduced due to the increase of oxygen content and reduce the carbon content. The increase of oxygen content would favor the complete combustion and reduce the production of black and particulate matter. The cloud and pour point properties of biodiesel is important especially in the cold places. The cloud point of biodiesel was found equal to 269 K and the pour point was found at value of 265 K. According to the specification ASTM D-6751-2, no restricted value was recorded. The flash point is an important safety property. The flash point for the biodiesel was found higher than the value of the standard biodiesel and mineral diesel. The acid value for the biodiesel of 0.38 mg KOH/g was found within the accepted value prescribed in the specification for the standard biodiesel.

TABLE II. THE PARAMETERS LEVEL FOR THE EXPERIMENTAL DESIGN

Properties	**Biodiesel**	**ASTM D 6751-02**	**diesel**	**ASTM Method**
Specific gravity	0.89	0.87–0.90	0.85	D4052
Viscosity at 313 K (mm^2/s)	5.4	1.9–6.0	1.9-4.1	D445
Calorific value (MJ/kg)	37.85	–	45	D2015
Pour point (K)	265	No value	-	D2500
Cloud point (K)	269	No value	261	D2500
Flash point (K)	448	403	325	D93
Acid value (mg/KOH/g)	0.38	0.8 max.	-	D664

IV. CONCLUSION

A direct conversion of sewage sludge oil was done in the microreactor device including the esterification and transesterification reactions using the DBSA as an efficient catalyst. A short space time have been consumed by the device for the conversion in comparison with the other types of reactors used industrially. An efficient design of experiment (Box-Behnken Design) was used for the optimization of the process due to the reduction in the number of experiments must be done. The optimum conditions for obtaining the maximum yield of biodiesel (experimental value=97.25, predicted value=97.75) were: reaction temperature (333.15 K), methanol/oil ratio (25:1), residence time (18 min), catalyst loading (7 wt%), co-solvent (16 wt %) for the microreactor system. The mixture of hexane-chloroform (50:50) as a co-solvent in the process shows a high activity for increasing the yield of biodiesel in comparison to the process of no-co-solvent. The physicochemical properties of the sewage sludge biodiesel produced in the microreactor system showed an accepted values in accordance to the biodiesel specifications ASTM D 6751-2 and the properties of the standard diesel oil.

ACKNOWLEDGMENT

We would lick to thank the University of Al Qadisiyah/College of engineering/Department of chemical engineering for providing the facility of the research.

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