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Continuous biodiesel production in a packed bed reactor from microalgae Chlorella sp. using DBSA catalyst

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ABSTRACT

Biodiesel production from Chlorella sp. oil using dodecylbenzenesulfonic acid catalyst in the packed bed reactor filled with a cylindrical glass raschig rings was investigated. The best conditions for the esterification and transesterification reactions were found to be (process temperature: 373.15 K, residence time: 30 min, methyl alcohol/oil ratio: 30, and catalyst loading: 11 wt%) which provide a yield of 99%. The water content effect was tested and the best value for the production of more than 90% biodiesel yield should be less than 0.5 wt%. Acid value was found to be decreased strongly using the DBSA catalyst in the esterification reaction. DBSA was found to be a highly active catalyst for the transesterification of Chlorella sp. compared to the activity of H_2SO_4 catalyst. The residence time of only 30 min using the DBSA catalyst would make the process more economical process compared to the batch process using sulfuric acid catalyst which consume more than 12 h. The flow characterization in the packed bed reactor was tested and characterized as laminar viscous flow with a Reynold number of 6.42×10^{-5}.

1. Introduction

In the last fifty years, the demand for energy was increased largely due to the vast growth of world population. Fossil fuels, the most fuel used nowadays caused many problems for the earth environment. As a result, the need for reducing the dependence on fossil fuels, led to the search for a new renewable sources of energy like biofuels. The types of biofuels have been classified as a first, second and third generation by research community. The first and second generation biofuels are either food crops or require large areas of land for cultivation. Microalgae, the third generation biofuel source is receiving a great attention as a biofuel for the reasons of the high growth rate, being not a source for food and has a smaller land footprint [22]. Many researchers have been focused on the transesterification of microalga using different catalysts and processes for biodiesel production. The best conditions for the two step process were investigated [24]. Direct transesterification of dry microalgae using alkaline catalyst was studied. The process was investigated with the addition of a different co-solvents to the reaction medium and/or using ultra sonication [19]. Direct lipase catalyzed transesterification of microalgae process was enhanced using ultrasound for reducing the time of reaction and increasing the yield of biodiesel [26]. Industrially, continuous packed bed reactor is a right choice for the production of biodiesel due to the enhancement of the reaction rate, direct product separation, reduction of energy and alcohol/oil consumption by intensifying the mass/heat phenomena. Commercially, PBR easily to be scaled up and used for the industrial production [23,29]. 3^3 FFD (full factorial design) and the BBD (Box-Behnken design) has been used in the optimization of the transesterification reaction for turnsole oil using a quicklime packed bed reactor. The BBD method was recommended for the optimization due to the smaller number of experimental runs. A yield of 99.1% was obtained at the best conditions [21]. Other researches have focused on the optimization of reaction parameters using the response surface methodology and factorial design. The microalgae species were directly changed to biodiesel using the CT-269 ion-exchange resin catalyst [30]. The fermented solids that containing lipases proved to be suitable for the conversion of fatty acids to esters with ethanol using the closed loop solid filled bed bioreactor [9]. Continuous homogenous catalyzed processes have been the subject of many papers concerning with the production of biodiesel using different alkaline and acid catalysts like NaOH, KOH, CH_3ONa and H_2SO_4 with a high yield value [15,3,6,16,27,25,13]. The catalytic activity of DBSA was

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compared to the other traditional acid catalysts used for the esterification and transesterification reaction like H_2SO_4, p-TSA and other catalysts with sulfonic groups. It was concluded that the increase in the hydrophopicity of the catalyst increases the reaction rate [1]. Morover, the DBSA was found to have a higher catalytic activity than other acid catalysts in the transesterification and esterification reactions under normal conditions [2]. In a recent review, the full scale and lab scale biodiesel production from microalgae were discussed extensively [11]. The extractive- transesterification of wet algae using 1-Butyl-3-methylimidazolium hydrogen sulfate was studied for evaluating the reaction parameters like the ratio of algae to catalyst, residence time and process temperature. The best high yield proved to be reached in a short residence time (30 min) [28]. To the best of our knowledge, there is no work have been carried out on the esterification and transesterification of micro algae using DBSA in a packed bed reactor. According to the above literature, using the DBSA as a catalyst for the esterification and transesterification of micro algae in a packed bed reactor would be useful industrially in the production of a potentially higher quantity of biodiesel of lower cost. The aims of the present study are, first to investigate the suitability of packed bed reactor for the creation of esters (biodiesel) using micro algae as a raw material and DBSA as a catalyst. Second, the process parameters were investigated (methyl alcohol/oil, catalyst loading, water content, process temperature, residence time) and their effect on the biodiesel and acid value were studied.

2. Experimental

2.1. Materials and measuring

Dodecylbenzenesulfonic acid (>0.99) was procured from Shanghai Hanhong Scientific Co., Ltd. Anhydrous $MgSO_4$ (99.8 wt %), chloroform (99.8), methyl alcohol (99.9%), ethanol (99%), hexane (98.8%) and gas chromatography (GC) standard methyl heptadecanoate for the quantitative analysis of FAME were provided from Sigma-Aldrich. All the chemical materials used were analytical reagents. Chlorella sp. extract was supplied from Shaanxi Jintai Biological Engineering Co., Ltd. The microalgae are dried at 343.15 K. The independent analysis of fatty acids shows that the lipid consists primarily from C16:0, C18:3, C18:2, C16:3, C16:2, and C18:0. The analysis of biodiesel produced at the best conditions for the free fatty acid profile and quantification were done using GC-2010-flame ionization detector (Shimadzu, Japan) armed with a split-splitless injector (oven temperature of 483.15 K, injection volume = 2 μL, split injection temperature of 513.15 K, split ratio = 1:50, flow rate = 1 ml/min, column specification of 30 m length, inside diameter = 0.25 mm, thickness of the film = 0.25, analysis time of 30 min and detector temperature of 523.15 K. Water content of the micro algae was tested with Karl Fischer tester (KF-1A). The acid value (mg KOH/g) of the oil extracted from microalgae was measured using the standard titration method according to the approved method of American Oil Chemists Society (AOCS). Each test was conducted in triplicate.

2.2. Extraction of oil

Extraction of oil was done in a 1000-mL round–bottom flask by mixing 200 g of dry Chlorella sp. with 600 ml of (chloroform/methyl alcohol of 1/1 vol) 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.

2.3. Continuous transesterification in a packed bed reactor

A direct transesterification was conducted using the filled bed reactor (internal diameter 2.5 cm, bed height 60 cm and total packed bed volume: 1178 cm^3 at normal pressure. The reactor temperature was kept under constant temperature using a water Jacket. The reactor was filled randomly by a porosilicate glass cylindrical packing. The schematic diagram for the experimental process system and the methodology were displayed in Fig. 1.

Methyl alcohol was mixed with a suitable amount of DBSA as a catalyst. Methyl alcohol-catalyst mixture and Chlorella sp. Oil was charged into the system using a dosing pumps. Before entering the packed bed reactor, methyl alcohol and Chlorella sp. Oil was preheated to 333.15 K. The parameters had been studied in the packed bed reactor and were as follows: process temperature (303.15 to 413.15 K), methyl alcohol/oil ratio (5–50), residence time (5–50 min), catalyst loading (1–19 wt%), and water content (0.5–6 wt%). The yield of biodiesel was investigated at different parameter condition and the experiments were repeated in triplicate (standard deviation was less than 5% for any point). The residence time inside the packed bed reactor was estimated using the subsequent equation [14]:

$$\tau = \frac{V_f}{v_o} = \frac{\pi.h.d^2.\beta}{4v_o} \quad (1)$$

where τ, V_f, υ_o, h, d, β are the residence time (min), volume of voidage (ml), volumetric stream rate (ml/min), height of packing in filled bed reactor (cm), inside diameter for the reactor (cm), and bed porosity, respectively.

The physical features of the Raschig rings used are provided in Table1. The cylindrical packing were weighed to determine the specific material density and by using the particle density and the bulk density, the porosity was determined. The glass Raschig ring's density was found to be 2.52×10^3 kg/m^3. 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 $MgSO_4$. GC-FID was used for the characterization of the product obtained from the packed bed reactor system. The tests were conducted in triplicate.

The yield of microalgae biodiesel was calculated according to the following equation:

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

where W_b, W_t, W_s, are the mass of biodiesel, total sample, and standard, respectively, A_b, A_s are the top area of biodiesel, and standard, respectively, CF_b, CF_s are the correction factor for biodiesel, and standard.

2.4. Filled bed reactor flow features

For flow characterization through the packed bed reactor, Reynolds number was calculated according to the following equation [22,18].

$$R_e = \frac{d_h.v.\rho}{\mu} \quad (3)$$

where d_h, υ, ρ, μ are the hydraulic diameter, fluid velocity (flow/cross section area of reactor), density of the flow mixture (890 kg/m^3), and fluid mixture viscosity (8.72×10^{-3} kg/m.s) respectively. The viscosity was measured at 1 am and 42 °C using Brookfield Viscometer (LVDVII-Canada).

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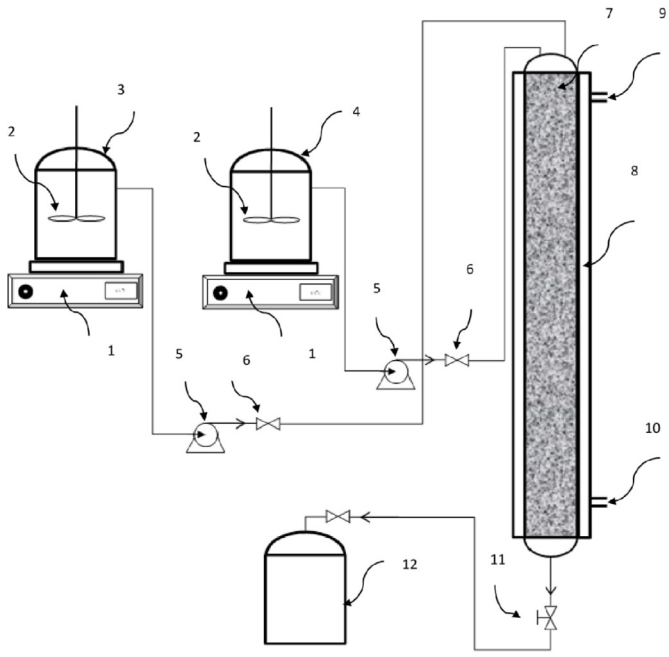

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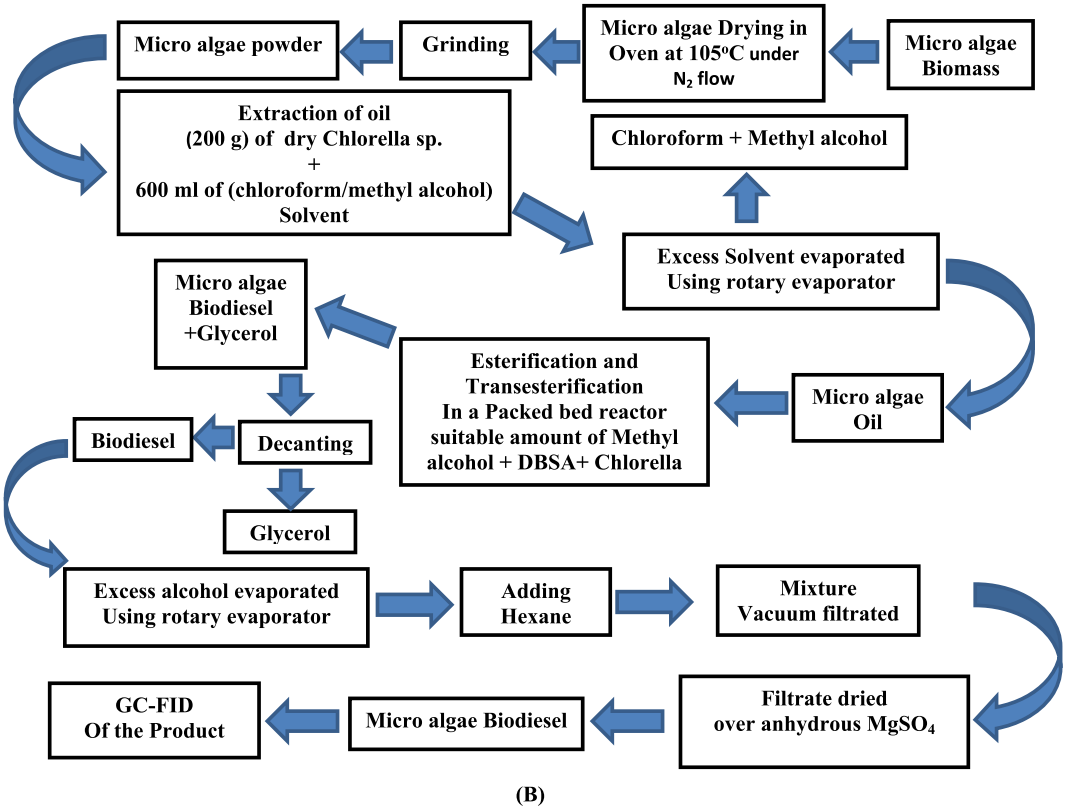

(B)

Fig. 1. (A) Representation figure of the filled bed reactor set-up: 1- Electrical heater; 2- mechanical mixer; 3- microalgae oil tank; 4- methyl alcohol tank; 5- dosing pump; 6- check valve; 7- rashig ring cylindrical packing; 8- water bath; 9- inlet water; 10- outlet water. 11- globe valve 12- biodiesel tank, (B) Schematic Diagram representing the methodology.

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Table 1
The physical properties of the packing material used in the present study.

Packing material	Material	Physical dimentions ($l \times b \times h$)	Particle volume (m^3)	Particle surface area (m^2)
Raschig rings	*Glass*	$0.007 \times 0.007 \times 0.009$	1.69×10^{-7}	3.7×10^{-4}

3. Results and discussion

3.1. Effect of methyl alcohol/oil

Effect of methyl alcohol/oil molar ratio is illustrated in Fig. 2. The transesterification and esterification reactions were carried out at a conditions of (process temperature: 373.15 K, residence time: 30 min, and catalyst loading: 5 wt% and 11 wt%). Many researches showed that biodiesel yield increase with increasing the mole percentage of methyl alcohol/oil due to favor the forward direction for the transesterification process [8,32,27]. Fig. 2 shows a small reduction of biodiesel yield for the ratio of methyl alcohol/oil of more than 30% due to the dissolving of biodiesel product in the excess methyl alcohol amount. Fig. 2 shows that the biodiesel yield at a catalyst loading of 11 wt% (biodiesel yield = 99%) was higher than that one at a 5 wt% (only 60%).

Fig. 3 shows the effect of methyl alcohol/oil molar ratio on the acid value at the same conditions mentioned above. The acid value exhibited a decrease from 5 to 0.5 wt% at a catalyst loading of 11 wt% when the methyl alcohol/oil ratio increased from 5 to 30 due to the conversion the FFA in the microalgae oil. At a catalyst ratio reaching 5 wt%, it is clear from Fig. 3 that the acid value was increased slightly due to the dilution in the concentration of DBSA catalyst at the ratio of methyl alcohol/oil more than 30. The same result was obtained in another study [31].

3.2. Effect of catalyst loading

For the investigation of the catalyst loading effect, the transesterification and esterification reactions were done in a packed bed reactor at a range of catalyst loading: 1–19 wt%, the ratio of methyl alcohol/oil:30, the residence time of 30 min and at a range of temperatures. Fig. 4 shows the effect of the catalyst loading on the biodiesel yield at three different temperatures. Obviously, Fig. 4 shows that the yield of biodiesel was increased considerably with rising the temperature from 338.15 to 373.15 K. The transesterification and esterification reactions favored to be done at higher temperature using an acid catalysts. Moreover, Fig. 4 shows that the yield of biodiesel was increased with the increase of the catalyst concentration from 1 to 19 wt% at 338 and 358 K. On the other hand, the biodiesel yield was increased to maximum value of 99% at a catalyst loading of 11% at a temperature of 373.15 K. Increasing the catalyst loading beyond the 11 wt% at a temperature of 373.15 K showed a small reduction in the yield of biodiesel. The increasing in the color darkness was observed at this reaction conditions. The reasons beyond the small reduction in the yield of biodiesel may be resulted from the carbonization in the presence of high concentration of DBSA. Many researchers stated the same conclusions in their works using sulfuric acid as a catalyst [7,5,20]. So that, the catalyst loading of 11 wt% was found the appropriate DBSA concentration for the transesterification and esterification reactions at the temperature of 373.15 K.

Fig. 5 shows the effect of the catalyst loading on the acid value at the temperatures of 358.15 and 373.15 K. Obviously, a similar scenario to that of the effect of methyl alcohol/oil ratio was obtained in the case of catalyst loading effect.

3.3. Effect of process temperature

The temperature is an important factor affecting the biodiesel yield. Fig. 7 shows the effect of the process temperature on the biodiesel yield at the reaction conditions of (catalyst loading: 11 wt%, ratio of methyl alcohol/oil: 30, residence time: 30 min for a range of temperature: 303.15–413.15 K). As expected, the biodiesel yield was increased with increasing the process temperature due to increase in the number of molecules of high kinetic energy. Further increase of temperature beyond 373.15 K would not further increase the biodiesel yield due to the endothermic behavior for the free fatty acids esterification process. Fig. 7 shows a slight decrease in the biodiesel yield due to the miscibility of biodiesel and glycerol, which favor the backward of the transesterification reaction.

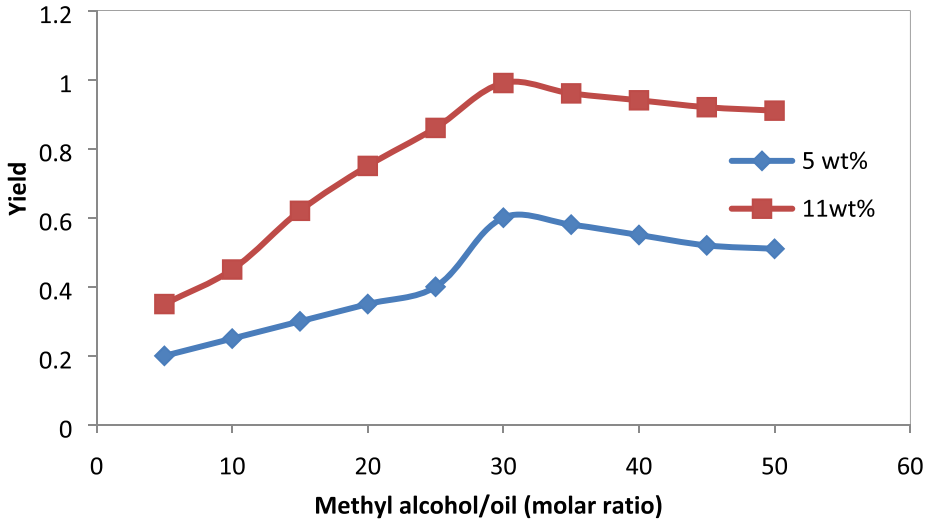

Fig. 2. Effect of the ratio of methyl alcohol/oil on the yield of biodiesel: process temperature 373.15 K; residence time 30 min; catalyst/oil 5 and 11 wt%.

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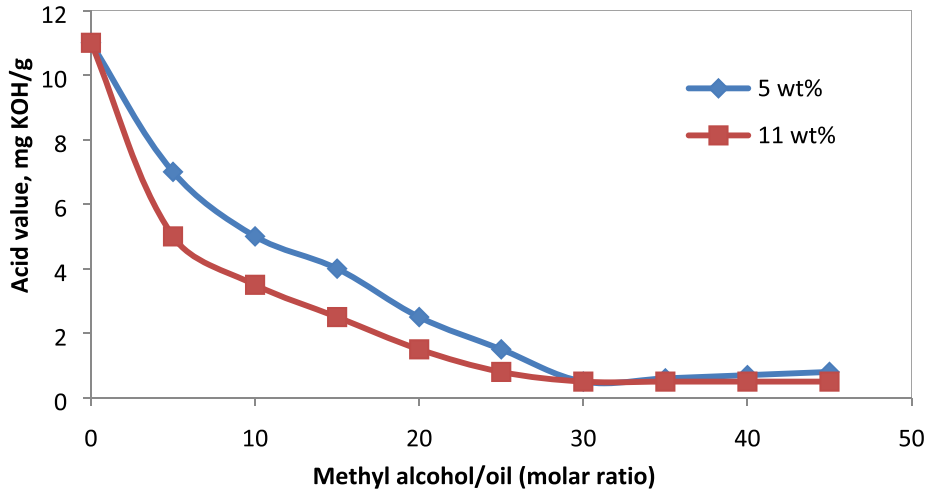

Fig. 3. Effect of the ratio of methyl alcohol/oil on acid value: process temperature 373.15 K; residence time 30 min; catalyst/oil 5 and 11 wt%.

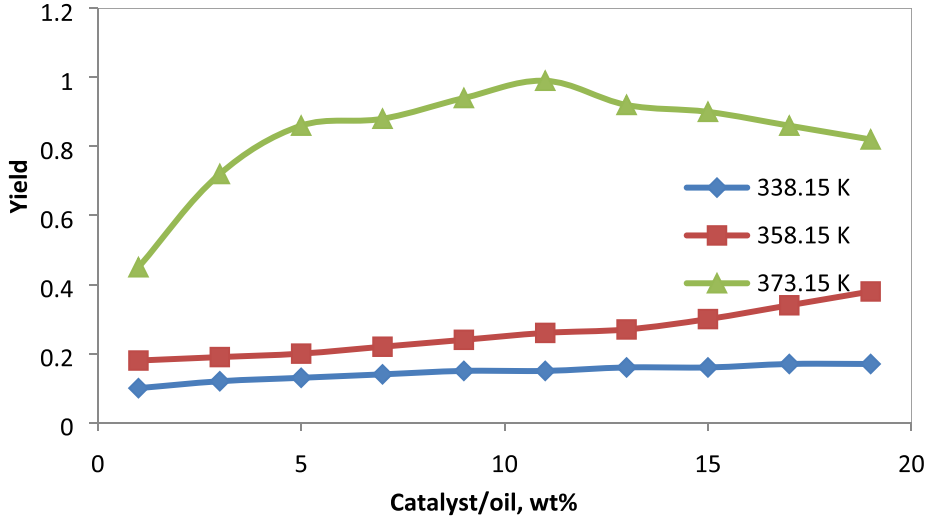

Fig. 4. Effect of the ratio of catalyst/oil on the yield of biodiesel: residence time 30 min; methyl alcohol/oil ratio 30; process temperature 338.15, 358.15, and 373.15 K.

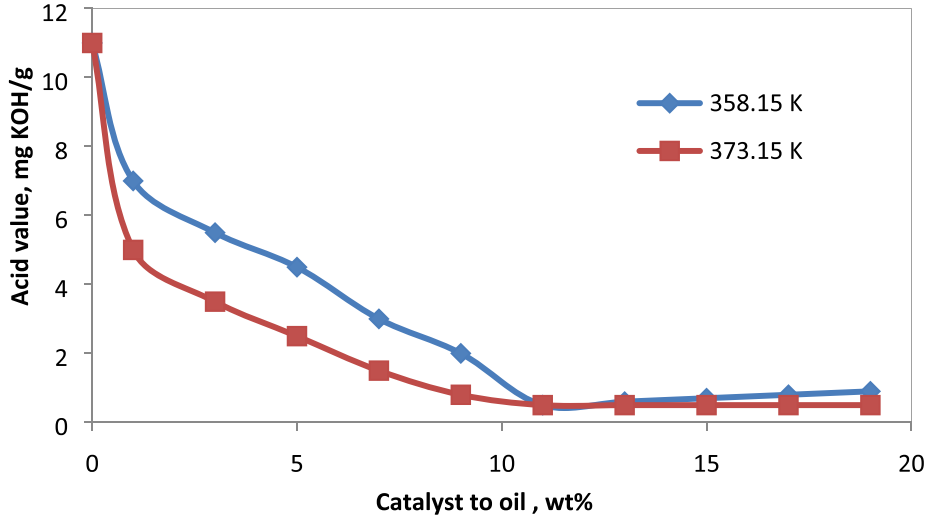

Fig. 5. Influence of catalyst/oil ratio on acid value: residence time 30 min; methyl alcohol/oil ratio 30; process temperature 358.15, and 373.15 K.

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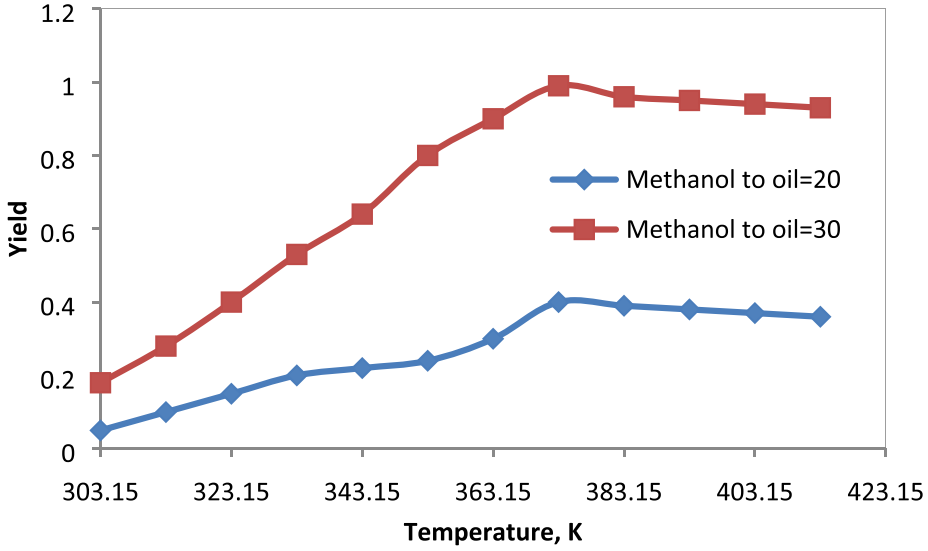

Fig. 6. Influence of process temperature on biodiesel yield: residence time 30 min; catalyst/oil 11 wt%; methyl alcohol/oil ratio 20 and 30.

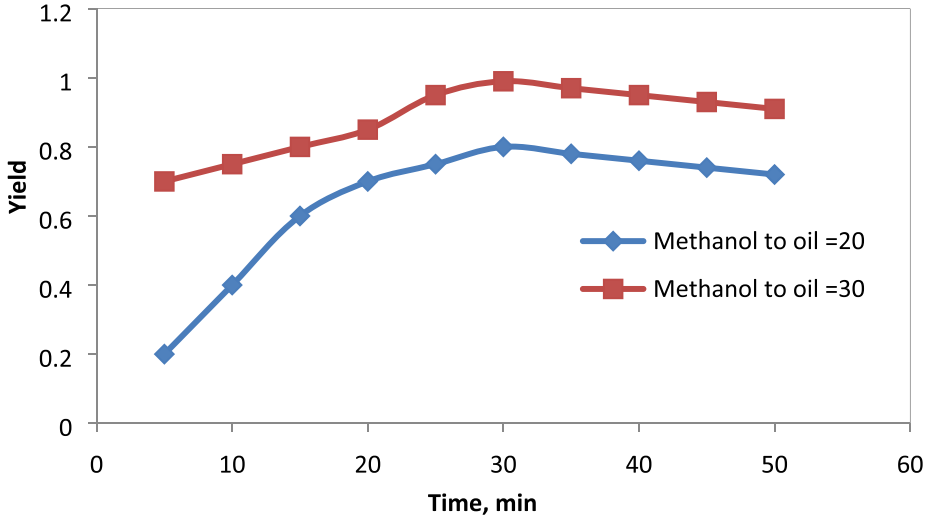

Fig. 7. Influence of residence time on biodiesel yield: process temperature 373.15 K; catalyst/oil 11 wt%; methyl alcohol/oil ratio 20 and 30.

3.4. Effect of residence time

The effect of residence time was studied by conducting an experiments at the reaction conditions of (catalyst loading: 11 wt %, methyl alcohol/oil ratio: 20 and 30, and process temperature: 373.15 K, and range of residence time of 5–50 min). Fig. 8 displays the influence of residence time on the yield of biodiesel at methyl alcohol/oil ratio of 20 and 30. Obviously, the yield of biodiesel got the extreme value at the residence time values extended between 10 and 30 min and exhibited a values of 88 and 99% at a methyl alcohol/oil ratio of 20 and 30. Beyond a residence time of 30 min, the figure shows a small reduction in the yield of biodiesel resulting from the reversible transesterification reaction which leads to the increase of di and monoglyceride molecules which was detected by GC. Increase of darkness was observed at longer residence times. As reported in another study, the same biodiesel yield was obtained from the sulfuric acid catalyzed transesterification of soybean oil at a longer time of 19 h [12]. The shorter time in packed bed reactor indicated the significance of packed reactors of the esterification and transesterification of microalgae oil.

3.5. Effect of water content

The influence of moisture content on the yield of biodiesel from microalgae oil was investigated by the addition of 1–6 wt% deionized water to the microalgae oil. Fig. 6 shows the effect of water content on the yield of biodiesel at the reaction conditions of (methyl alcohol/oil ratio: 30, catalyst loading: 11 wt%, residence time: 30 min, and a process temperature: 373.15 K). Biodiesel yield was decreased suddenly to less than 5% with increasing the water content from 1 to 8 wt%. The negative influence of moisture content on the yield of biodiesel also was concluded in other researches using H_2SO_4 catalyst for the transesterification of rapeseed and grease [4,17].

3.6. Flow in the filled bed reactor

The characterization of flow in the filled bed reactor was evaluated using Reynolds number (Eq. (3)). The flow was characterized as laminar when Renolds number ($\leq$ 2000) and turbulent when Renolds number ($\geq$ 4000). The value of the Renolds number in

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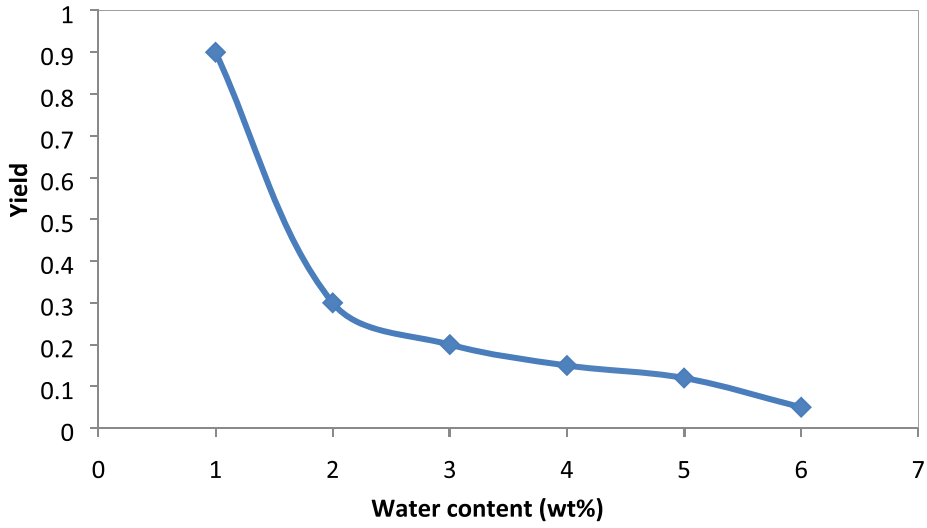

Fig. 8. Influence of water content on biodiesel yield: process temperature 373.15 K; catalyst/oil 11 wt%; methyl alcohol/oil ratio 30; residence time 30 min.

the present study was found (6.42×10^{-5}) which indicates the laminar behavior of the flow and the flow dominants by the viscous flow. E. Silva et al. also obtained the laminar flow characterization at different flow rates for the biodiesel production of babassu and macaw palm oils with ethanol in a packed bed reactor [10]. The significance of the Reynolds number can be explained importantly in the case of scaling up the packed bed reactor for industrial scale as follows. As the flow is laminar, the selectivity of the reaction can be correlated using viscous regime and a special expressions can be developed depending on the Reynolds number for the scale-up of diameter and length of the reactor.

4. Conclusion

The water content of microalgae should be decreased to less than 0.5 wt% in order to obtain a conversion of in excess of 90% in the packed bed reactor. Based on the conditions of the process, DBSA was found to be an efficient catalyst for the esterification and transesterification reactions. The residence time of the process was only 30 min using the DBSA catalyst and would make the process more economical process compared to the batch process using sulfuric acid catalyst which consume more than 12 h. The biodiesel yield in the filled bed reactor was found to be 99% at the best conditions of the process. The hydrophobic structure of the DBSA makes it suitable catalyst for the esterification reaction and reduce the formation of soap and proved in the present study as an excellent catalyst for the transesterification reaction. The massive commercial production of DBSA makes it the right choice in the packed bed reactor. The laminar viscous flow provided by the packed bed reactor was providing a suitable contact between the oil, methyl alcohol and DBSA. The flow was characterized by the value of (6.42×10^{-5}) Reynolds number. Based on the present work results, it is recommended to scale up and develop the process for the industrial level using the suitable special expressions.

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