Advances in Energy Research, *Vol. 8, No. 4(2022) 233-241*
https://doi.org/10.12989/eri.2022.8.4.233

Optimized biodiesel yield in a hydrodynamic cavitation reactor using response surface methodology

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(*Received July 25, 2021, Revised August 23, 2022, Accepted September 2, 2022*)

Abstract. Biodiesel is a non-polluting and non-toxic energy source that can replace conventional diesel. However, the higher production cost and raw material scarcity became challenges that obstruct the commercialization of biodiesel production. In the current investigation, fried cooking oil is used for biodiesel production in a hydrodynamic cavitation reactor, thus enhancing raw material availability and helping better waste oil disposal. However, due to the cavitation effect inside the reactor, the hydrodynamic cavitation reactor can give biodiesel yield above 98%. Thus, the use of orifice plates (having a different number of holes for cavitation) in the reactor shows more than 90% biodiesel yield within 10 mins of a time interval. The effects of rising temperature at different molar ratios are also investigated. The five-hole plate achieves the highest yield for a 4.5:1 molar ratio at 65°C. And the similar result is predicted by the response surface methodology model; however, the optimized yield is obtained at 60°C. The investigation will help understand the effect of hydrodynamic cavitation on biodiesel yield at different molar ratios and elevated temperatures.

Keywords: biodiesel; free fatty acid; fried cooking oil; hydrodynamic cavitation; orifice plate

1. Introduction

Petroleum products have become a necessity for a country's development. However, the pollution from burning petroleum products is a matter of concern as the earth's temperature is rising faster due to greenhouse gases. In contrast, biodiesel is a good energy source (Mourad *et al.* 2021, Singh *et al.* 2020, Uddin *et al.* 2013) and can be produced from both usable and non-usable oils, thus, making it a renewable source of energy (Goh *et al.* 2020, Singh *et al.* 2020). Biodiesel has properties closer to conventional diesel, making it the best competitor to diesel (Kachhwaha *et al.* 2010). It is a non-polluting fuel, absorbing an equal amount of CO_2 by oilseed plants generated during fuel combustion in the engine (Kumar *et al.* 2013). However, biodiesel has slightly less energy than diesel, which reduces about 10-15% engine performance in existing diesel engines, while the emissions decline significantly (Mourad *et al.* 2021, Palani *et al.* 2020). Thus, developing engines exceptionally for biodiesel fuel will overcome this problem, and in this process, further reduction is viable in engine emissions.

Biodiesel is produced from raw oils and animal fats. The raw oil has triglycerides that react

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http://www.techno-press.org/?journal=eri&subpage=7 ISSN: 2287-6316 (Print), 2287-6324 (Online)

with alcohol molecules to produce biodiesel and glycerin. The use of catalysts reduces reaction time and enhances the biodiesel yield while maintaining a specific reaction temperature range; this reaction is called a transesterification reaction (Jayakumar *et al.* 2021, Lapuerta *et al.* 2009). However, raw material scarcity is always a problem in biodiesel production because edible oil can never be a good option. In contrast, the waste fried cooking oil (FCO) disposal from restaurants and hotels is a critical challenge in metro cities (Goh *et al.* 2020). Thus, the researchers have studied the potential of FCO in biodiesel production and found that the transesterification of waste FCO produces biodiesel with similar properties as from other sources (Uddin *et al.* 2013). The transesterification process significantly lowered the density and viscosity of FCO, thus making the FCO biodiesel properties closer to conventional diesel (Dehghani *et al.* 2019, Intarapong *et al.* 2016). The methods used for the transesterification process include mechanical stirring, ultrasonic cavitation, hydrodynamic cavitation, and microwave technique.

Mechanical stirring is a time-consuming method and gives up to 90-92% biodiesel yield, while ultrasonic cavitation gives biodiesel yield of 97-98% with about 45 minutes reaction time, and hydrodynamic cavitation lies between both with more than 90-95% biodiesel yield (Kumar *et al.* 2013). However, hydrodynamic cavitation has the potential to raise biodiesel yield by raising the cavitation effect. In cavitation, fluid atomizes into smaller oil droplets and develops tiny cavitation bubbles. In the turbulent zone, these bubbles collapse, generating shock waves. The shock waves help improve oil-alcohol interaction, triggering the transesterification reaction (Park *et al.* 2008).

The existing compression ignition (CI) engines are designed for diesel fuel, while biodiesel has slightly higher physio-chemical properties than diesel (Miron *et al.* 2021). As discussed earlier, the transesterification process reduces some of the physio-chemical properties of raw oil that transforms into biodiesel; however, biodiesel-diesel blending is still required to match engine fuel specifications as per ASTM standards (Mourad *et al.* 2021). The biodiesel-diesel blends show comparatively lower emissions, with the expense of engine performance, while ethanol addition in blends significantly enhanced engine performance. The ethanol blending provides extra oxygen for better fuel combustion (Lapuerta *et al.* 2009, Li *et al.* 2005). The result showed that about 5 to 10% ethanol concentrations improved engine performance significantly (Al-Hassan *et al*. 2012, Barabás *et al.* 2010) and reductions in emissions when alcohol (methanol, ethanol) is used in biodiesel-diesel blending (Barabás *et al.* 2010). The literature showed a 30% reduction in particulate matter and a 5.6 to 11.4% decline in NO_X emissions due to higher exhaust temperature (Shi *et al.* 2006). Other emissions like CO and HC are lowered significantly due to better fuel combustion (Labeckas and Slavinskas 2013).

In this work, the cavitation effect of different orifice plates (having different holes) is determined to maximize the biodiesel yield in a hydrodynamic cavitation reactor for the transesterification of fried cooking oil (FCO). Using FCO as raw material will help in sustainable disposal to save the environment from its adverse effects, and in the process, FCO showed an excellent potential source for biodiesel production. Further, the results of the molar ratio at elevated temperatures are investigated. A response surface methodology model is prepared to optimize the yield parameters.

2. Materials and experimental setup

2.1 Materials

Fried cooking oil (FCO) is gathered from the college campus canteen and filtered for any solid suspended impurities. Alcohol for transesterification reaction is industrial-grade methyl alcohol in different molar ratios with the oil. The literature showed a higher biodiesel yield with a 1:6 molar ratio (Ong *et al.* 2021). While KOH pellets, having a molecular weight of 56.11, is used as a homogeneous catalyst for the reaction. Another alkyl alcohol used to prepare biodiesel-diesel-ethanol blends is an industrial-grade ethyl alcohol AR 99.9%. The different parameters considered are shown in Table 1.

2.2 Hydrodynamic cavitation reactor

A hydrodynamic cavitation reactor comprises a reaction chamber of a 12-liter feed tank with a control valve, as shown in Fig. 1. The filtered raw FCO and the mixture of KOH catalyst in methanol are poured through the inlet opening. The Inlet valve is shut, and the reciprocating pump of 7.36 kW, working in a closed-loop circuit, is switched ON. The external heater is used to maintain the temperature in the range from 55℃ to 65℃. The circulation is allowed for 10 min. time interval before the samples are taken from the drain opening. A pressure gauge and flow meter are installed to monitor the fluid pressure and flow rate (a mixture of raw FCO, methanol, and KOH) within the reaction chamber.

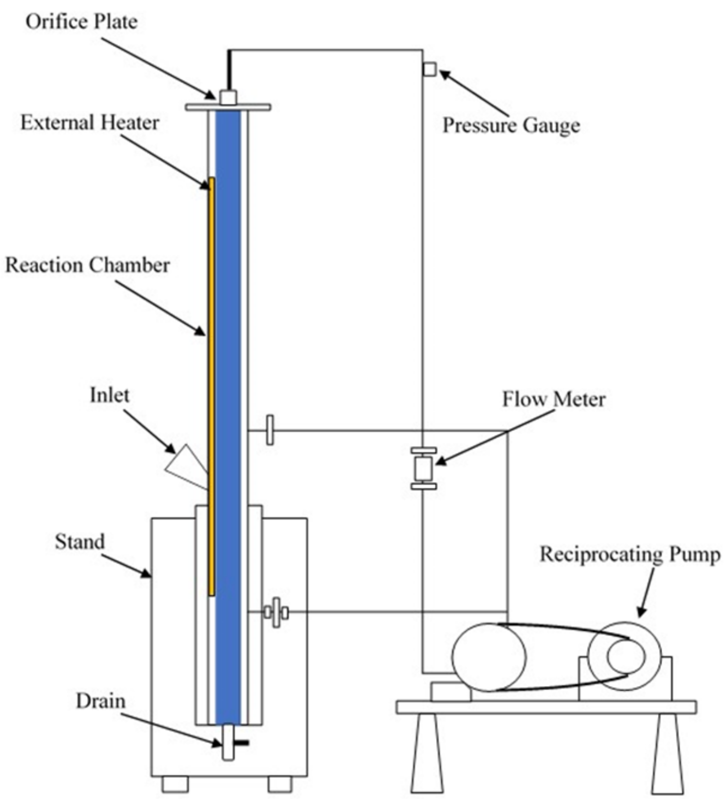

Fig. 1 Hydrodynamic cavitation reactor

Table 1 Parameters and levels for biodiesel optimization

Parameters	Levels		
	1	2	3
Molar ratio	3:1	4.5:1	6:1
No. of holes	1	3	5
Reaction Temperature	55°C	60°C	65°C

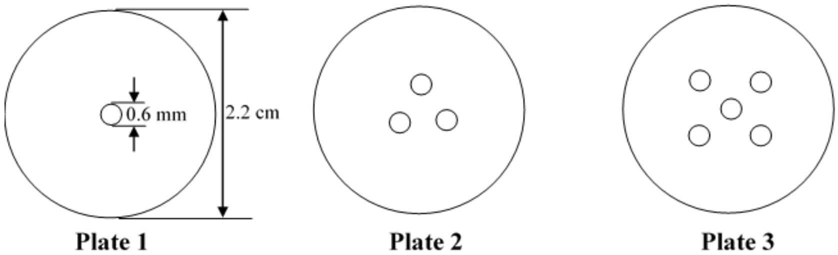

Fig. 2 Orifice plates with different numbers of holes

The three orifice plates with a diameter of 2.2 cm and a hole diameter of 0.6 mm are used to maximize the cavitation effect in the reaction chamber (Fig. 2). All the plates have different holes, as shown in Fig. 2, i.e., plate 1 with one hole; plate 2 with three holes; and plate 3 with five holes to boost the reactor's cavitation effect.

3. Procedure

Fried Cooking Oil (FCO), gathered from the DTU campus canteen, is filtered and separated from any solid suspended impurities left after frying. Initial heating removes any moisture content in the raw oil because the moisture content may result in saponification, reducing biodiesel yield. Thus, the initial heating of 3 batches of 6 kg raw FCO each is performed at 110°C for about 10 minutes, and the oil is allowed to cool down at room temperature. The free fatty acid (FFA) content of FCO is measured and required to determine because FFA content of more than 2.5% requires an esterification process (making the whole process two-stage transesterification). Thus, 2.14% of FFA content concludes in a single-stage transesterification after the titration method.

Potassium hydroxide (KOH) is initially dissolved in methanol before adding to the raw FCO. About 60 gm of KOH (1 wt.%) is dissolved in 500 gm of methanol using a magnetic stirring process and then poured into the hydrodynamic cavitation reactor. Additional methanol is added to the hydrodynamic cavitation reactor to make a 3:1 molar ratio. The catalyst concentration (1 wt.%) is chosen from earlier literature work (Ma and Hanna 1999, Mohite *et al.* 2016, Singh *et al.* 2020). The orifice plate 1 is mounted to the hydrodynamic cavitation reactor, and the reciprocating pump is switched ON. An external heater maintains the temperature between 55°C and 65°C depending upon the experiment run. The circulation is allowed for a particular time interval before taking the

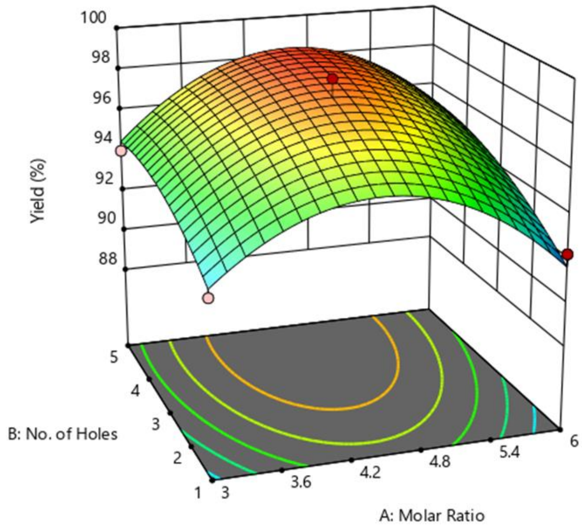

Fig. 3 Molar ratio vs. number of holes (in plate) surface-contour plot

samples from the drain opening, and three samples are taken after 10 min. The average of 3 samples is considered for forming the research surface methodology (RSM) model.

The samples are stored uninterrupted for the next 24 hours to settle down the heavy glycerine under the action of gravity. The layers of biodiesel mixed with KOH and glycerine become visible after the settlement. The above layer consists of biodiesel taken out, and water washing removes catalyst KOH from biodiesel. In the water washing process, biodiesel containing KOH is poured into a separating funnel, and one-third by volume of warm water (at 50-60°C) is added and stored undisturbed for 4-5 hours. The water-KOH mixture will settle down and thus be removed easily. The left-out biodiesel is heated to 110°C for 5-10 mins to remove moisture present after water washing.

4. Results and discussion

4.1 Biodiesel yield

Maximum biodiesel yield is the object in the transesterification reaction. The parameters like reaction temperature, molar ratio, catalyst concentration, catalyst type, and reaction time can be varied to optimize biodiesel yield. Here, the molar ratio, reaction temperature, and the number of holes on the plate (that cause the cavitation effect) are taken for optimization in this experimental work. The optimization of various parameters is studied using response surface methodology (RSM), and their effects on biodiesel yield are discussed briefly.

4.1.1 Effect of molar ratio and number of holes

The molar ratio is an essential parameter in biodiesel yield that affects the conversion efficiency of raw oil and production cost (Ma and Hanna 1999). A lower molar ratio results in an

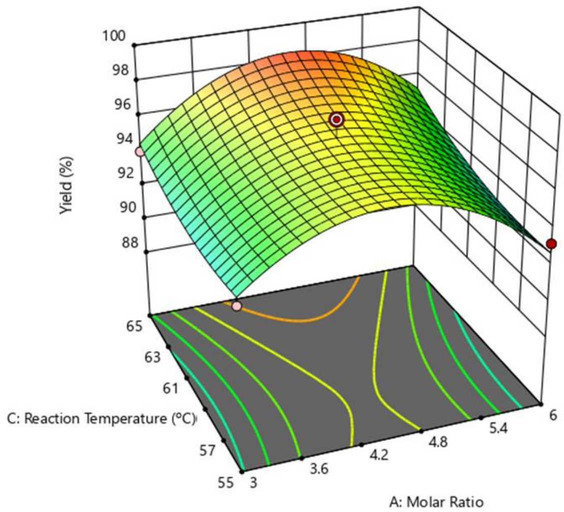

Fig. 4 Molar ratio vs. number of holes (in plate) surface-contour plot

incomplete reaction, while the higher molar ratio complicates glycerol separation, thus, resulting in reduced biodiesel because a fraction of the glycerol remains in the biodiesel phase (Lee and Saka 2010). Therefore, optimizing the molar ratio will raise the biodiesel yield and reduce the production cost. The number of holes to molar ratio effect on biodiesel yield is shown in Fig. 3. It is clear that lower and higher molar ratio reduces the biodiesel yield significantly. Also, a greater number of holes on the plate results in more cavitation making better oil-to-alcohol interaction. Earlier studies show that a 9:1 to 15:1 molar ratio is good for transesterification (Ma and Hanna 1999, Singh *et al.* 2020). However, the hydrodynamic cavitation has reduced the optimized molar ratio to 4:1 to 5:1, and the highest yield is achieved at 4.5:1 molar ratio with plate having five-holes.

4.1.2 Effect of molar ratio and reaction temperature

Along with the molar ratio, reaction temperature also plays a vital role in conversion rate and production cost. At lower temperatures, the reaction becomes slow and thus, takes more time for completion, while higher temperature reduces the reaction time (Halwe *et al.* 2021). From Fig. 4, it is clear that increasing reaction temperature increases the biodiesel yield, and the maximum biodiesel yield is achieved between a 4:1 to 5:1 molar ratio. The cavitation effect allows more yield at a higher temperature because as the temperature rises, the cavitation effect also rises, making the better oil-to-alcohol interaction. Olubunmi *et al.* (2022) reported that a reaction temperature of 60°C is optimal for higher biodiesel yield.

4.1.3 Effect of number of holes and reaction temperature

As discussed, the number of holes raises the cavitation effect, and higher temperature favors cavitation. The result in Fig. 5 shows that as the temperature increases, the biodiesel yield increases, and similarly, the plate with five holes gives more biodiesel yield than a plate with a lower number of holes. However, the optimization technique shows that a plate with three holes optimizes biodiesel yield.

Table 2 Biodiesel sample properties

Blends	Density (g/cm^3)	Specific gravity	Kinematic viscosity	Calorific value (MJ/kg)	Cetane index
Diesel	0.8346	0.8372	3.4416	45.14	48.1
Ethanol	0.7892	0.7916	1.5142	29.62	12
Biodiesel (B100)	0.9053	0.9082	6.2145	38.08	42.3
B20	0.8491	0.8517	3.9964	43.74	46.7
B10E10	0.8379	0.8406	3.5271	42.91	43.8

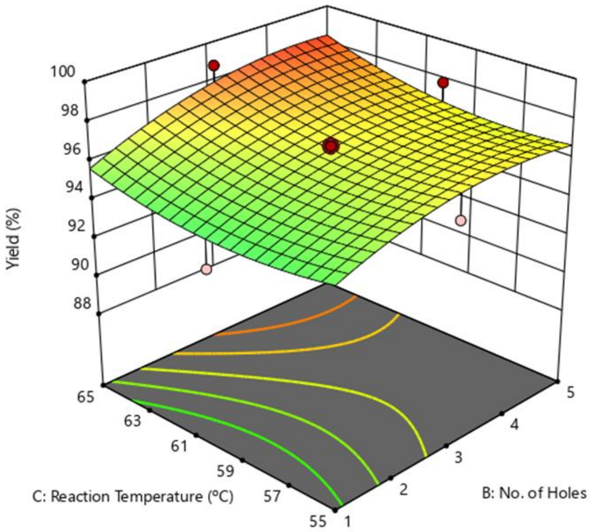

Fig. 5 Reaction temperature vs. number of holes (in plate) surface-contour plot

4.2 Fuel properties

Diesel is collected from the nearby gas station, and the density (ρ), specific gravity, kinematic viscosity, and calorific value (CV) are measured as 0.8346 g/cm^3, 0.8372, 3.4416 mm^2/s, and 45.14 MJ/kg. While ethanol is more volatile than other fuels, it has a density of 0.7892 g/cm^3, a specific gravity of 0.7916; kinematic viscosity of mm^2/s; and CV of 29.62 MJ/kg. Biodiesel is denser than diesel and has 0.9053 g/cm^3 density, 0.9082 specific gravity, 6.2145 mm^2/s kinematic viscosity, and 38.08 MJ/kg calorific value, and these values are very close to diesel fuel as computed in Table 2.

5. Conclusions

In this work, biodiesel is prepared from the transesterification of fried cooking oil (FCO) and methyl alcohol with catalyst potassium hydroxide (KOH) in a hydrodynamic cavitation reactor. Three orifice plates of 0.6 mm hole diameter are used. Each plate has a different no. of holes that show different cavitation effects inside the reactor. Further, molar ratio and temperature are also varied to understand their influence on the cavitation effect. The following outcomes are found in this work: -

- The maximum biodiesel yield of 98.77% is achieved by plate 2 with three holes at a 4.5:1 molar ratio and 65℃.
- The optimized biodiesel yield of 96.8% is obtained using the RSM model.
- The optimized parameters are a 4.5:1 molar ratio, a plate with five holes, and a 60℃ reaction temperature.
- Biodiesel properties are measured and found closer to conventional diesel.

The experimentation showed that waste FCO has excellent potential for biodiesel production, and the hydrodynamic cavitation method may produce as high as 98% biodiesel yield. Therefore, implementing the hydrodynamic cavitation process increases biodiesel yield above 90%, and the temperature and molar ratio also helps raise the yield percentage further. Future work may involve determining the effect of different hole sizes and increasing the no. of holes.

Acknowledgments

Department of Mechanical Engineering, Delhi Technological University, Delhi, India, supported this work. No funding is received for the above work.

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