



Intensified synthesis of biodiesel using hydrodynamic cavitation reactors based on the transesterification of waste cooking oil



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HIGHLIGHTS

- Hydrodynamic cavitation results in significant process intensification.
- Waste cooking oil is a good sustainable feedstock for transesterification.
- Slit venturi is the best cavitating device.
- Better properties of the final synthesized biodiesel.

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ABSTRACT

In the present work, hydrodynamic cavitation reactor has been used for the intensification of synthesis of biodiesel from waste cooking oil (WCO) based on the transesterification reaction. Experiments have been performed using different cavitating devices such as orifice plate, circular and slit venturi and under varying operating parameters viz. inlet pressure (2–5 bar), molar ratio of oil to methyl acetate (over the range of 1:10–1:14) and catalyst loading over the range of 0.5–1.25% by weight of oil. Maximum yield (90%) of biodiesel from WCO was obtained at oil to methyl acetate ratio of 1:12 and catalyst loading of 1.0% using slit venturi at the inlet pressure of 3 bar. Also, higher cavitation yield (biodiesel produced per unit energy consumed) was obtained for the hydrodynamic cavitation based approach as compared to the ultrasound based approach and conventional method. The observed intensification is attributed to the microscale turbulence generated due to the cavitation effects that help in intensifying the transfer processes. Overall, the potential of WCO to produce good quality methyl esters in energy efficient manner using hydrodynamic cavitation reactor has been demonstrated based on the cavitation yield calculations and properties of synthesized biodiesel.

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1. Introduction

Enormous increase in the fuel consumption due to increase in the population and industrialization has resulted in considerable stress on the fossil fuel production, also having significant impact on economies of many countries. The use of petroleum based products creates substantial air pollution problems all over the world [1] and different alternatives are being explored currently to develop synthesis routes for alternate fuels such as biodiesel. Biodiesel is similar to the petroleum based diesel in many aspects and can be synthesized by chemical reaction involving any oil or fat with alcohol. The sources of oil or fat can vary from sustainable sources such as animal fats, waste cooking oil, non-edible oils, and agricultural residues to food grade vegetable oils. Biodiesel has advantages over

petroleum based diesel such as biodegradability, non toxic nature, high flash point, excellent lubricity, higher cetane number, no sulphur content and lesser pollution [2,3]. Biodiesel can be used neat (complete replacement) or as a diesel additive and hence has a good potential to replace the fossil fuel [4]. Apart from the advantages of biodiesel, there are some limitations to the use of biodiesel as a fuel and most important one is the higher cost of production. The cost of biodiesel is almost two times as compared to the cost of petroleum based diesel fuel [5], which is typically attributed to higher costs of feedstock [6], lower conversion rates and higher reaction time as well as significant costs associated with the downstream separations. The production cost can be reduced by using sustainable feedstock such as WCO and also based on the use of transesterification reaction instead of transesterification. Transesterification reaction between oils/fats and methyl acetate [7] results in the formation of triacetin, which has higher value (significant use as a plasticizer, a gelatinizing agent in polymers and as an additive in

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pharmaceutical, tobacco and cosmetics industries) as compared to the glycerol produced in transesterification. Triacetin can also be blended with biodiesel up to 20% by weight matching the quality standards set by ASTM D6451 [8].

Interesterification has been mostly studied so far using the enzymatic route [9–11] or based on the supercritical conditions [12–15], but both these approaches offer significant disadvantages. On one side where the enzymatic approach require considerable processing time and higher production costs [15,16], the supercritical methods require significantly high pressures (20–40 MPa) and temperatures (350–400 °C) as well as significant excess of methyl acetate (usually reactant ratio of oil to methyl acetate is set at 1:42) [13]. Overall, it can be said that though interesterification based approach is good for synthesis of biodiesel with a value added product, use of process intensifying approach is required for possible reduction in the production cost and for enabling the use of ambient conditions. Cavitation reactors can be used for intensification of interesterification reaction very similar to more deeply investigated transesterification approaches. There are also other approaches for biodiesel production such as microwave, oscillatory baffle reactors, microreactors, and supercritical method [1,17] as well as the use of reactive distillation [18]. The rate of biodiesel production is controlled by the mass transfer limitations associated with the reactant phases and the physical effects of cavitation such as intense turbulence (generated at microscale) and streaming can be effective in eliminating these resistances. These effects are responsible for generation of fine emulsions which provide large area for reaction leading to intensified operations. Indeed, cavitation reactors have been used for the intensification of the biodiesel production from waste cooking oil [19], palm fatty acid [20] and Nagchampa oil [21] though based on the esterification/transesterification reactions, however the present work is the first attempt for the application of hydrodynamic cavitation for the intensification of interesterification based synthesis of biodiesel. Cavitation effects can be produced using two routes of acoustic cavitation (passage of high intensity ultrasound through the liquid medium) and hydrodynamic cavitation (passage of liquid at high flow rates through a constriction). When the liquid passes through the constriction, there is a sudden increase in the velocity of liquid at the expense of the local pressure and if this local pressure falls below the vapour pressure, number of cavities will be generated that subsequently collapse with the recovery of pressure downstream of the mechanical constriction [22–24]. Hydrodynamic cavitation has been widely used for the wastewater treatment or esterification/transesterification reactions, but there has been absolutely no study reporting the use of hydrodynamic cavitation for intensification of interesterification based synthesis of biodiesel. The current work reports the novel study of intensification of interesterification reaction of pretreated waste cooking oil in the presence of potassium methoxide using hydrodynamic cavitation. Potassium methoxide has been selected on the basis of results presented by Casas et al. [7] for the interesterification reaction where the effect of type of catalyst was investigated using different catalysts such as potassium hydroxide, potassium methoxide and polyethylene glycolate. It was reported that higher biodiesel yield is obtained in the presence of potassium methoxide and hence potassium methoxide has been used in the present work instead of more commonly used potassium hydroxide or sodium hydroxide. Experiments have been performed using different geometries of cavitating device and effect of inlet pressure, methyl acetate to oil molar ratio and catalyst concentration on the biodiesel yield has also been investigated. Kinetic analysis for the interesterification reaction has also been presented with estimation of the properties of synthesized biodiesel.

2. Materials and methods

2.1. Materials

Methyl acetate, potassium methoxide pellets, ortho phosphoric acid and molecular sieves (3°A) were procured from S.D. Fine Chem. Ltd., Mumbai whereas weak anion-exchange resin (Indion 860) was obtained from Ion Exchange Ltd., Mumbai. Acetonitrile and acetone (HPLC grade) used as solvent for HPLC analysis were obtained from Hi Media, Mumbai whereas methanolic potassium methoxide (33% by wt.) was procured from Spectrochem Pvt. Ltd., Mumbai. The standards of methyl oleate and methyl linoleate were obtained from Sigma Aldrich. Waste cooking oil (WCO) was procured from a local restaurant and was subjected to initial physical treatment and repeated water washing as per the details provided in the earlier work [1]. The properties of treated WCO used as the starting raw material have been given in Table 1. As the initial acid value of the WCO was high at 3.89 which is unsuitable for use in the presence of alkaline catalyst, deacidification of WCO has been performed. Weak anion exchange resin (Indion 860, 1 wt.%) has been used for the removal of free acid content under conditions of stirring at 800 rpm and 273 K where the acid value was reduced from 3.89 to 0.4 in 3 h of treatment.

2.2. Reactor configuration

The schematic of the experimental setup is shown in Fig. 1. The basic setup is similar to that used in the earlier work [25], but the present work also uses different geometries of cavitating device such as slit venturi, circular venturi in addition to the more commonly used orifice plate. The setup consists of a holding tank of 15 L volume, a multistage centrifugal booster pump of power rating 1.5 kW and a recirculation loop provided with control valves, main line which accommodates the cavitating device and a bypass line to control the flow. The range of inlet pressure used in the work was 1–5 bar with the inlet flow rate varying over the range 1.58–5.83 LPM ($2.63 \times 10^{-5} \text{ m}^3/\text{s}$ to $9.72 \times 10^{-5} \text{ m}^3/\text{s}$). The pressure gauge P1 is used to measure the inlet pressure of the liquid. In this system, the reactants flow continuously from the tank to cavitating device and is introduced back into the tank (recirculation mode of operation) using a centrifugal pump. Due to these effects, it is expected that uniform mixing of the reactants occurs in the tank. The recirculation through the cavitation chamber is intensifying the reaction due to the cavitation effects leading to the creation of fine emulsion and generating large interfacial area for the reaction. Depending on the increase in the flow rate, the number of passes experienced by the reactant mixture through the cavitation chamber will increase. For example, when the flow rate is at minimum of 1.58 LPM, the number of recirculation of the entire reactant mixture would be around 6 during the entire

Table 1
Physical and chemical properties of waste cooking oil.

| Property | Value |
|--|-------|
| <i>Physical</i> | |
| Saponification value (mg KOH/g of oil) | 207.7 |
| Density (kg/m ³) | 928 |
| Acid value (mg KOH/g oil) | 3.89 |
| Viscosity (mm ² /s) | 96.5 |
| Water content (%) | 0.7 |
| <i>Chemical</i> | |
| Linoleic acid (%) | 73.4 |
| Oleic acid (%) | 18.3 |
| Palmitic acid (%) | 6.7 |
| Stearic acid (%) | 1.6 |

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