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Intensified production of biodiesel using a spinning disk reactor

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ABSTRACT

This work achieves continuous transesterification of soybean oil and methanol in a spinning disk reactor. The effects of the methanol-to-oil molar ratio, catalyst type, catalyst concentration, reaction temperature, flow rate, and rotational speed were investigated. Optimal yield of 96.9% was obtained with a residence time of 2–3 s at a molar ratio of 6, potassium hydroxide concentration of 1.5 wt%, temperature of 60 °C, flow rate of 773 mL/min, and rotational speed of 2400 rpm. The production rate of 1.86 mol/min was high compared to that of other reactors for continuous transesterification process, indicating that a spinning disk reactor is a promising alternative method for continuous biodiesel production.

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1. Introduction

Biodiesel, an alternative fuel, is produced from vegetable oils and animal fats. Most commercial biodiesel is made by transesterification, which transforms triglyceride and methanol into fatty acid methyl esters (FAMEs) in the presence of alkaline or acid catalyst. However, some problems are associated with this process. First, conventional reactors for biodiesel production are typically operated in batch mode, which is labor intensive and high cost, yielding poor production efficiency. As a result, a continuous production process is preferred by industry. In 2000, Darnoko and Cheryan [1] performed continuous transesterification of palm oil using a continuous stirred-tank reactor (CSTR). Potassium hydroxide (KOH) was the catalyst. Experimental results showed that a yield of methyl esters of 97.3% can be achieved with a residence time of 60 min.

Another limitation of current biodiesel production is the significant mass transfer resistance between the oil and alcohol because both are immiscible [2]. Consequently, reaction time is usually long when biodiesel is produced in a conventional stirred tank due to its low mixing and mass transfer efficiency. The long reaction time decreases throughput or manufacture must increase reactor size. To overcome these problems, several novel reactors have been developed to accelerate the mixing of vegetable oil and methanol [3]. In 1998, Noureddini et al. [4] used two mixers, first a motionless mixer and then a high-shear mixer, to convert soybean oil into methyl esters with sodium hydroxide as a catalyst. Conversion was 98% with a residence time of 6.67 min. In 2007, Stavarache et al. [5] proposed a continuous process for manufacturing biodiesel from commercial edible oil under ultrasonic irradiation. Conversion exceeded 90% with a residence time of 10 min. In 2008, Sun et al. [6] produced biodiesel in capillary microreactors with unrefined rapeseed oil and cottonseed oil as raw materials and KOH as the catalyst. When the methanol-to-oil molar ratio was 6 and the catalyst concentration was 1%, FAMEs yield was over 95% with a residence time of roughly 6 min.

Recently, more reactors have been investigated for continuous production of biodiesel with a short residence time. In 2009, Kraai et al. [7] used sunflower oil and methanol to produce biodiesel in a centrifugal contactor separator. Optimum conversion was 96% with a residence time of 11 min when the catalyst (sodium methoxide) concentration was 1 wt%, the molar ratio of methanol to oil was 6, and the oil feed rate was 12.6 mL/min. In 2010, Chen et al. [8] produced biodiesel from soybean oil and methanol in a rotating packed bed with KOH as the catalyst. Conversion was 97.3% with a residence time of 0.73 min when the catalyst concentration was 3 wt% and reaction temperature was 60 °C. In 2011, Santacesaria et al. [9] studied transesterification of soybean oil with methanol catalyzed by KOH in a tubular reactor filled with small spheres. With a residence time of 1 min, conversion was 98% when the catalyst concentration was 2 wt% and reaction temperature was 60 °C. To produce biodiesel from canola oil and methanol with sodium hydroxide as the catalyst, Lodha et al. [10] utilized a rotating tube reactor. When rotational speed was 670 rpm, conversion was 97.65% with a residence time of 0.75 min. Phan et al. [11] transesterified rapeseed oil with methanol in oscillatory baffled reactors. Conversion was 97% with a residence time of 5 min, KOH

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concentration of 1.5%, and methanol-to-oil molar ratio of 6. Transesterification of canola oil with methanol in an intensified spinning disk reactor was achieved by Qiu et al. [12]. The reactor comprises two flat disks separated by a small gap. The upper disk rotates and the lower disk remains stationary. The feed streams flow radially outward in the gap. Conversion was related to the gap width, rotational speed, feed flow rate, and surface topology of the disk. Conversion was about 87% at a rotational speed of 1000 rpm and reaction temperature of 60 °C with a very short residence time (<1 s). In 2013, Karanja oil and methanol were used to produce biodiesel in a helical tube reactor using KOH, as the catalyst by Agarwal et al. [13] Maximum conversion was 92.6% when residence time was only 4 min.

In 2006, Chen et al. [14] reported that a spinning disk reactor (SDR) provided the highest mixing efficiency of several mixing devices, including a continuous flow stirring reactor, a Couette flow reactor, a static mixer, an ultrasound-assisted flow cell, and a rotating packed bed. An SDR consists of a rotating disk within a stationary housing. The liquid enters at the disk center and flows rapidly outward as thin films on the disk surface. The SDR is used for polymerization and production of micro- or nano-particles by precipitation because of its remarkable micromixing efficiency. Boodhoo and Jachuck [15] performed free-radical polymerization of styrene in an SDR. The reaction rate was enhanced significantly, when the molecular weight distribution was small. In 2006, Boodhoo et al. [16] studied cationic polymerization of styrene in an SDR. Compared to a conventional stirred batch reactor, polymerization in an SDR can use a higher monomer concentration and higher reaction temperature because an SDR has excellent heat transfer efficiency. Nano-size $Mg(OH)_2$ particles were synthesized in an SDR by Chen et al. [14]. Increasing micromixing intensity reduced particle size. Moreover, silver nanoparticles were synthesized in an SDR via a green chemical process, using environmentally friendly and inexpensive glucose and starch as the reducing agent and protecting agent, respectively [17]. In 2010, Chen et al. [18] recrystallized p-aminosalicylic acid particles using an anti-solvent precipitation process in an SDR. Drug particles with a mean size of 1 μm were obtained and the dissolution rate of micronized drug particles was enhanced significantly.

In this work, an SDR was the reactor for continuous biodiesel production. Transesterification of soybean oil with methanol using KOH or sodium methoxide ($NaOCH_3$) as a catalyst was carried out in an SDR. Experimental conditions, such as the methanol-to-oil molar ratio, catalyst concentration, reaction temperature, rotational speed, and flow rate were varied. The yield and production rate of biodiesel obtained in this SDR were compared to those obtained in other reactors.

2. Experimental

Soybean oil was purchased from the Taiwan Sugar Corporation. 99.9% pure methanol was purchased from the Fisher Chemical Company. 95% pure KOH was purchased from the Showa Chemical Company. 95% pure $NaOCH_3$ was purchased from the Acros Organic Company. 99.8% pure isopropyl alcohol was purchased from Scharlau; and 96% pure sulfuric acid was purchased from Sigma–Aldrich. The FAMEs standards, which included methyl palmitate, methyl stearate, methyl oleate, methyl linoleate, and methyl linolenate, were purchased from Merck.

Fig. 1 displays the main structure of the SDR. The main parts were a stainless-steel disk driven by a motor and a stationary cylindrical chamber. System temperature was controlled by recirculating water inside the disk and chamber. The SDR was 14 cm in diameter. The disk rotational speed was varied from 300 to 2400 rpm. The liquid distributors were two straight tubes in parallel and the radial distance of each distributor was 1 cm from the disk axis. Both tubes had a 3-mm hole at the end that was placed 5 mm from the disk.

In this study, biodiesel was produced by transesterification of soybean oil with methanol in an SDR. The KOH and $NaOCH_3$ were used as a catalyst, respectively. Soybean oil and methanol solution containing the catalyst were placed in the constant-temperature bath. The desired reaction temperature was achieved by heating the disk and stationary cylindrical chamber with recirculating water. The two reactants were then pumped continuously at a specific flow rate into the SDR by peristaltic pumps. The two liquid streams from the distributors entered at the disk center and flowed rapidly outward by centrifugal force as a thin film on the disk surface. Consequently, a very high shear force and mixing effect were achieved. The flow then hit the stationary chamber and exited through the reactor bottom. The collected sample (10 mL) was immediately mixed with a 0.25 M sulfuric solution to stop the reaction. The upper layer of the resulting mixture was then washed with warm water to remove the catalyst and other impurities. A gas chromatography analyzer (GC 1000; China Chromatography) equipped with a FID and a capillary column (Wax10; Supelco) was used for product analysis. Samples were prepared by dissolving 0.5 ml of product in 25 ml isopropyl alcohol. Nitrogen was the carrier gas. The oven temperature program started at 170 °C, and increased at 2 °C/min to 200 °C. The injector and detector temperatures were 250 and 260 °C, respectively. The yield of FAMEs of the transesterification process is calculated as

$$\text{Yield } (\%) = \frac{M_{\text{FAMEs}}}{M^0_{\text{FAMEs}}} \times 100 \quad (1)$$

where M_{FAMEs} is the mass of FAMEs in the sample. M^0_{FAMEs} is the mass of FAMEs in the samples if the oil is completely converted and is approximately equal to the mass of initial oil. The production rate of FAMEs (P) is defined as

$$P = 3 \times \text{yield} \times N_{\text{oil}} \quad (2)$$

where N_{oil} is the molar flow rate of oil.

3. Results and discussion

Continuous transesterification of soybean oil with methanol was achieved in an SDR. Samples were taken from the outlet stream after the system reached a steady state (within 10 min). The variation in concentration measurements was ±5%. The yield and production rate of FAMEs under various experimental conditions are shown in Table 1. Fig. 2 shows the effect of the methanol-to-oil molar ratio on yield of FAMEs. Methanol-to-oil molar ratios of 3–12 were investigated at a fixed flow rate of 267 mL/min, temperature of 60 °C, rotational speed of 1800 rpm and 1.5 wt% KOH. Experimental results show that maximum yield was achieved at a molar ratio of 6. The stoichiometric ratio for transesterification requires 3 moles of methanol and 1 mole of oil. However, transesterification is a reversible reaction; a high molar ratio is used to shift the chemical equilibrium toward the desired products. Additionally, increasing the methanol-to-oil ratio enhances the solubility, reduces the viscosity and increases the amount of contact between methanol and oil molecules. However, excess methanol acts as an emulsifier and makes the separation of esters from methanol and glycerol difficult. Consequently, a methanol-to-oil molar ratio of 6 was used in the following experiments.

Fig. 3 shows the effect of catalyst concentration on yield of FAMEs. Two alkali-catalysts, KOH and $NaOCH_3$, with concentrations of 0.5–3 wt%, were investigated at a fixed methanol-to-oil molar ratio of 6, temperature of 60 °C, rotational speed of 1800 rpm, and flow rate of 267 mL/min. Experimental results show that FAMEs

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