



## A new co-solvent method for the green production of biodiesel fuel – Optimization and practical application

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### HIGHLIGHTS

- ▶ The transesterification reaction can be performed at room temperature ( $25 \pm 1$  °C).
- ▶ Reduced time for separation of glycerin (within 30 min after reaction completion).
- ▶ Reduced reaction time (less than 30 min).
- ▶ Reduction of a significant methanol amount (molar ratio of methanol to oil, 4.5:1).
- ▶ High FAME yield.

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### ABSTRACT

An homogeneous reaction process using acetone as a co-solvent for the transesterification of vegetable oils with methanol in the presence of potassium hydroxide catalyst has been developed. The effects of the main parameters, such as the amounts of acetone and KOH, the molar ratio of methanol to oil and the reaction temperature, on fatty acid methyl esters (FAME) yield were investigated. The optimal conditions were: 25 wt.% acetone and 1.0 wt.% KOH catalyst in oil; a molar ratio of methanol to oil of 4.5:1; and a reaction temperature of 25 °C. Under these conditions, the conversion of vegetable oil to FAME exceeded 98% after 30 min. This method was applied to produce biodiesel fuel (BDF) from waste cooking oil (WCO), canola, catfish and *Jatropha curcas* oils, with the main product quality factors satisfying the JIS 2390 BDF standards.

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### 1. Introduction

Currently, transportation is almost totally dependent on fossil liquid fuels such as gasoline, diesel, and kerosene. The price of petroleum has been unpredictable and significantly increased in recent years. Furthermore, the excessive use of fossil fuels has had negative impacts on the environment, including air pollution, global warming, and climate change. Therefore, it is necessary to find alternative fuels that may help to sustain liquid fuel use, while improving the quality of the atmospheric environment. In this context, biofuels, in particular biodiesel and bioethanol, are appropri-

ate renewable options for overcoming the limits of fossil-based fuels [1–3].

Conventionally, biodiesel is produced via the transesterification reaction of triglyceride (the main component of vegetable oils and animal fats) with an alcohol, usually methanol or ethanol, in the presence of suitable catalysts such as acid, base or enzymes, to form mono-alkyl esters. The type of catalyst used depends on the amount of free fatty acid (FFA) and water present in the raw oils. Homogeneous acid catalysts are preferred for oils containing a high concentration of FFA, i.e. 5 wt.%, because the reaction does not form soap [4]. However, the disadvantages of acid catalysts are that the reaction rate is 4000 times lower than that of base catalysts and the reaction requires a high molar ratio of methanol to oil, e.g. 12:1, and long reaction times, e.g. 48 h [4,5]. In contrast, homogeneous base catalysts give high FAME yields within short reaction times under mild reaction conditions. However, homoge-

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neous base catalysts require high-quality raw materials that preferably contain low concentrations of FFA and water, leading to increased production costs.

Biodiesel production costs are highly dependent on the raw materials, and the cost of vegetable oils or animal fats is usually a major contributor to the overall cost, at about 70% [6]. Biodiesel produced from virgin vegetable oils costs much more than petrodiesel, and this is a major barrier to the commercialization of biodiesel. Therefore, it is necessary to identify cheaper raw oils to minimize the production cost of biodiesel. From this point of view, the use of WCO and non-edible oils such as catfish, *Jatropha curcas*, rubber, and tobacco oils are attractive options for reducing the cost of raw materials [7–11].

Several methods for biodiesel production, including mechanical stirring, supercritical alcohol, and microwave processes have been developed [12–14]. However, to reduce the cost of production processes and to mitigate adverse effects on the environment, innovative methods that require less raw materials and consume less energy have been reported. In previous work, we have developed sonication processes for FAME production [15,16]. In the sonication method, high quality biodiesel was produced with minimal material costs and energy consumption. However, the method still requires a significant time, about 4 h, for the phase separation of glycerin (GL) from the reaction mixture. As a result, the overall time required for the complete process is prolonged [17].

To overcome the difficulties listed above, in previous work, we developed an homogeneous reaction process using co-solvents such as acetone, 2-propanol, tetrahydrofuran, or ethyl acetate for the transesterification of several oils with methanol in the presence of KOH catalyst. From this work, acetone was found to be the best co-solvent for biodiesel production because the production process can be carried out under mild conditions, at room temperature (25 °C) and with a brief separation time of 30 min [18].

In this study, the optimal reaction conditions for transesterification using acetone as a co-solvent were identified. These conditions were subsequently applied successfully to produce biodiesel from canola, catfish and *Jatropha curcas* oils at the laboratory scale and used as design parameters to build a pilot plant.

## 2. Experimental

### 2.1. Materials

Canola oil was purchased from the Nisshin Oil Company, Tokyo, Japan. WCO was from domestic use, collected by municipal authorities, and then filtered and settled to remove solid materials. Catfish oil was purchased from An Giang province and *J. curcas* from Binh Thuan province, Vietnam. The chemical and physical properties of these oils are listed in Table 1. KOH grade 95.5%, ace-

tone and acetonitrile (both HPLC grade) were purchased from Wako Pure Chemical Industries, Osaka, Japan. Chemical standards such as triolein, diolein, monoolein and methyl oleate were obtained from Sigma Aldrich (St. Louis, MO, USA) and used without further purification.

### 2.2. Procedure

#### 2.2.1. Transesterification of oil and purification of FAME

Transesterification was performed in a 100 mL Erlenmeyer flask immersed in a water bath equipped with a thermostat to maintain the desired reaction temperature. For each experiment, 40 g of oil was premixed with a certain amount of acetone with a magnetic stirrer bar for 30 s until the mixture formed a single phase and then a solution of methanol and KOH catalyst was added. Thereafter, the reaction mixture was gently stirred for 30 min to allow the reaction to proceed, then settled for 1 h to allow phase separation. After the completion of phase separation, acetone, mainly remaining in the FAME phase, was recovered by a rotary evaporator system (EYELA SB-1100) at 60 °C under a reduced pressure of 50.6 kPa for 30 min. The crude FAME was transferred into a 125 mL funnel, where impurities were removed by washing three times with 50 °C distilled water (at a ratio of water to FAME of 20 wt.%). The pH values of the FAME and the water layers were in the range of 6–7 after the third washing. After washing, the water remaining in the FAME was effectively eliminated by heating the FAME phase to 80 °C under a reduced pressure of around 53.3 kPa for 1 h.

### 2.3. Analysis

To investigate the concentration of reactants and products such as triglycerides (TG), diglycerides (DG), monoglycerides (MG) and FAME during the reaction processes, 3 mL samples were withdrawn at specific times from the reaction mixture and transferred into a 10 mL glass vial containing 2 mL of 5% phosphorus acid aqueous solution to stop the reactions. The samples were left to settle for 2 h for phase separation. The upper phase contained primarily acetone, FAME, TG, DG and MG, and the lower phase contained excess methanol, GL, potassium phosphate and water. The upper phase was removed and heated to 70 °C for 2 h to remove acetone. 40 µL of the FAME phase was sampled and the exact mass measured and it was then diluted in 4 mL of acetone solvent for high performance liquid chromatography (HPLC) analysis.

The concentrations of FAME, TG, DG and MG were quantified by gel permeation chromatography (GPC). The GPC apparatus consisted of a pump (Shimadzu, LC-10AD) connected to a column (Asahipak GF-310 HQ, 300 × 7.5 mm) and refractive index detector (Shimadzu, RID-10A). The column oven was fixed at 30 °C. The mobile phase was acetone, at a flow rate of 0.5 mL/min and the sample injection volume was 20 µL. Calibrations were carried

**Table 1**  
Chemical and physical properties of canola, WCO, catfish and *Jatropha curcas* oils used in this study.

Properties	Unit	Oils (average ± SD <sup>b</sup> )			
		Canola	WCO	Catfish	<i>Jatropha curcas</i>
Density	g cm <sup>-3</sup>	0.915 ± 0.002	0.918 ± 0.002	0.916 ± 0.003	0.913 ± 0.003
Acid value	mg KOH/g oil	0.80 ± 0.01	1.07 ± 0.02	4.23 ± 0.02	6.67 ± 0.03
Oleic acid (C18:1) <sup>a</sup>	wt.%	61.4 ± 0.5	47.0 ± 0.6	27.2 ± 0.5	43.8 ± 0.7
Linoleic acid (C18:2) <sup>a</sup>	wt.%	22.3 ± 0.6	31.4 ± 0.4	46.7 ± 0.5	31.7 ± 0.5
Linolenic acid (C18:3) <sup>a</sup>	wt.%	11.5 ± 0.4	10.2 ± 0.4	3.4 ± 0.3	3.7 ± 0.6
Stearic acid (C18:0) <sup>a</sup>	wt.%	2.5 ± 0.3	2.8 ± 0.3	12.0 ± 0.3	6.0 ± 0.3
Palmitic acid (C16:0) <sup>a</sup>	wt.%	1.2 ± 0.3	7.4 ± 0.3	8.4 ± 0.3	12.7 ± 0.5
Other fatty acids	wt.%	1.1 ± 0.2	1.2 ± 0.2	2.3 ± 0.3	2.1 ± 0.3

<sup>a</sup> Carbon atoms number: double bonds number.

<sup>b</sup> SD: Standard deviation.

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