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 ± 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.

ARTICLE INFO

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 curcus* 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 Oillio 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 triglyceries (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[b])			
		Canola	WCO	Catfish	*Jatropha curcas*
Density	g cm^{-3}	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)[a]	wt.%	61.4 ± 0.5	47.0 ± 0.6	27.2 ± 0.5	43.8 ± 0.7
Linoleic acid (C18:2)[a]	wt.%	22.3 ± 0.6	31.4 ± 0.4	46.7 ± 0.5	31.7 ± 0.5
Linolenic acid (C18:3)[a]	wt.%	11.5 ± 0.4	10.2 ± 0.4	3.4 ± 0.3	3.7 ± 0.6
Stearic acid (C18:0)[a]	wt.%	2.5 ± 0.3	2.8 ± 0.3	12.0 ± 0.3	6.0 ± 0.3
Palmitic acid (C16:0)[a]	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

[a] Carbon atoms number: double bonds number.
[b] SD: Standard deviation.

out using chemical standards such as triolein, diolein, monoolein and methyl oleate for quantification of TG, DG, MG, and FAME.

The concentrations of fatty acids in the oils in the FAME phase, including oleic, linoleic, linolenic, palmitic, and stearic acids, were quantified by HPLC using a Cadenza C-18 column (250×3 mm) with the refractive index detector. The analytical method was described previously in more detail [16].

3. Theory

Several reviews dealing with the production of biodiesel by transesterification have been published [19,20]. Commonly, transesterification is catalyzed by a base or an acid catalyst and TG is converted stepwise to DG and MG intermediates, and finally to GL [21]. The transesterification of TG with alcohol in the presence of a base catalyst obeys the S_N2 mechanism [22].

Because methanol and oil are immiscible, transesterification occurs in a heterogeneous system at the interface between the component layers and, as a result, the reaction rate and the yield of FAME are low. To solve this problem when using the conventional mechanical stirring method, the reaction is usually carried out at the boiling point of methanol to increase mass transfer between methanol and oil. Another approach is to use ultrasound emulsification, in which the reaction mixture is subjected to low frequency ultrasound in the range of 20–40 kHz to produce a fine emulsion, leading to an increased interfacial area between the reactants. However, the reaction still takes place only at the interface between the immiscible reactants present in the heterogeneous phases [23,24]. It has been suggested that the addition of solvents such as tetrahydrofuran, diethyl ether, or 1,4-dioxane into the reaction mixture can increase dissolution of methanol into the oil, creating a homogeneous reaction system. The reaction can thus be accelerated through molecule–molecule reaction and can be completed in a shorter reaction time, with an increased FAME yield [25,26].

In previous work, acetone was found to be the best solvent for the production of biodiesel [18]. As shown in Table 2, although acetone has a large dipole moment of 2.88 D, it is classified as an aprotic solvent, with intermediate polarity. Therefore, it can be dissolved well in both TG (low polarity) and methanol (high polarity). Furthermore, an important property of acetone, as an aprotic solvent, is its ability to stabilize the methoxide ion, CH_3O^-, which is the active intermediate for the transesterification reaction, according to the S_N2 mechanism [27]. Acetone can therefore effectively increase the transesterification reaction rate. The physical and chemical properties of oil and FAME are dependent on the fatty acid composition and distribution in the oil and FAME. Therefore, triolein and methyl oleate, which are the main components of oils and FAME, present representative properties for TG and FAME. The physical properties of the solvent and reactants are given in Table 2.

4. Results and discussion

To determine the optimum conditions for the production process, the transesterification of WCO with methanol was conducted in the presence of KOH catalyst. The effects of the main reaction parameters, such as the amounts of acetone and catalyst, the molar ratio of methanol to oil and the reaction temperature on FAME yield were examined.

4.1. Effect of acetone amount

The amount of acetone is one of the most important factors and it directly affects the FAME yield and the time required for separation of GL from the reaction mixture. The purpose of this experiment was to find the minimum amount of acetone required to accelerate the reaction in the homogeneous system and shorten the separation time of GL from the reaction mixture. The amount of acetone was varied over the range 0–30 wt.%, based on the WCO weight.

Fig. 1 shows that the FAME yield significantly increased with increasing acetone addition. At acetone concentrations below 20 wt.%, the reaction mixture did not become homogeneous and FAME formation was slow. After a 60 min reaction, the FAME yields attained were 55.3%, 74.3%, 83.5% and 88.3% corresponding to acetone concentrations of 0, 5, 10 and 15 wt.%, respectively, and the reaction continued to proceed beyond 60 min. In contrast, the reaction mixture became homogeneous after 40, 32 and 20 s and the FAME yields were 92.6%, 96.1% and 96.1% at 20, 25 and 30 wt.% acetone, respectively. As presented in our previous study, transesterification requires a longer time, i.e. 50 min, in the case of the heterogeneous system, because the reaction occurs at the interface between droplets of oil and methanol [16]. However, in the homogeneous system, reaction takes place between TG and methanol at the molecular level, leading to a higher FAME yield and shorter reaction time.

Furthermore, the addition of acetone reduces the time required to separate GL from the mixture after completion of the reaction, as described previously [18]. Photograph 1 displays the difference in the GL layer formed from the reaction mixture after settling for 30 min with differing amounts of acetone. It was observed that after 60 min reaction, the times required for GL separation were 37, 30 and 50 min for acetone contents of 20, 25 and 30 wt.%, respectively. The separation time was defined as the time at which the GL phase was completely separated from the reaction mixture and two clear phases were formed (the GL and the FAME phases), as shown in the vessel containing acetone 25 wt.% in Photograph 1. The FAME phase remained turbid when the acetone content was below 20 wt.% because the reaction was not complete and a number of small droplets of methanol and GL remained in the FAME phase. Generally, as the amount of acetone increases, the viscosity of the reaction mixture decreases and the difference between the densities of the FAME and GL phases increases, resulting in a reduced time required for phase separation [18]. Contrary to expectations, a further increase in the amount of acetone to 30 wt.% led to an increase in the time required for the phase separation. This behavior can be explained as follows: the separation of GL and excess methanol in the reaction mixture is dependent on their concentrations. With a lower acetone content, the concentrations of GL and methanol are higher than with a larger acetone content. Therefore, the probability of collisions between GL and methanol is increased so they readily collide to rapidly develop a GL phase and separate from the reaction mixture. This finding agrees with

Table 2
Physical properties of solvent and reactants used in this study [27–30].

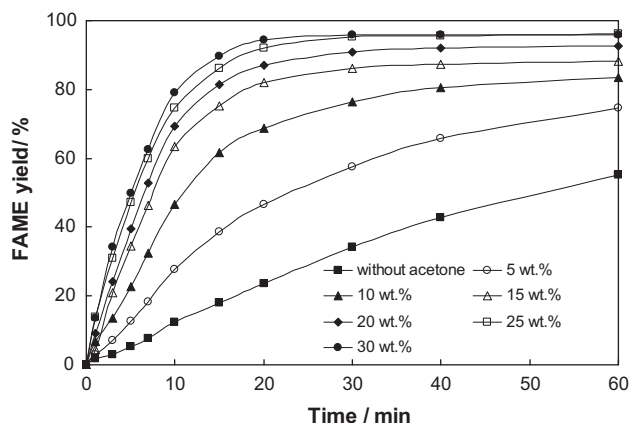| Name	Boiling point (°C)	Density at 20 °C (g/mL)	Dielectric constant (at 25 °C)	Dipole moment
Acetone	56	0.786	20.7	2.88 D
Methanol	65	0.791	32.6	1.70 D
Triolein	235	0.912	3.2	–
Methyl oleate	218 at 20 hPa	0.872	–	–
GL	290	1.261	42.5	2.66 D

Fig. 1. Effect of the amount of acetone on the FAME yields. The reaction conditions: molar ratio of methanol to WCO, 5:1; KOH catalyst, 0.5 wt.%; temperature, 25 ± 1 °C.

the result of Issaiyakul et al. 2010, who found that the addition of a large amount of GL (20 wt.% relative to oil) after reaction separated the GL layer from the FAME layer within 1 h [31].

4.2. Effects of KOH catalyst content

The transesterification reaction is catalyzed by alkaline metal hydroxides or alkoxides, as well as sodium or potassium carbonates. The alkaline catalysts provide good performance when high quality raw materials (FFA < 0.5 wt.% and moisture < 0.5 wt.%) are

used [32]. In general, the reaction is carried out at 60–65 °C under atmospheric pressure with excess methanol. Several studies have suggested that KOH or NaOH and their methoxides act as effective catalysts for the transesterification, with typical concentrations from 0.4 to 2 wt.% of oil [33]. We recently reported that methanolysis of WCO with 1.0 wt.% KOH assisted by ultrasound gave the best yield of FAME (99%) [17].

In this study, the effect of KOH on the formation of FAME was investigated at concentrations ranging from 0.1 to 1.25 wt.%, with a constant methanol to oil molar ratio of 5:1. Fig. 2 shows the conversions of FAME plotted against reaction time.

Fig. 2 indicates that the best yields of FAME, obtained at catalyst concentrations higher than 1.0 wt.%, were greater than 98%. Maximum FAME yields were reached after 20 and 30 min at catalyst concentrations of 1.25 and 1.0 wt.%, respectively, but did not reach 97% at lower catalyst concentrations, even after 60 min. This can be explained as follows. The transesterification reaction begins by the methoxide ion (CH_3O^-) attacking the carbonyl carbon atoms of TG, DG, and MG molecules [34]. Because KOH is a strong base, it dissociates extensively in methanol to form CH_3O^- and therefore, the amount of CH_3O^- depends on the catalyst concentration. As a result, a larger amount of KOH catalyst increases the reaction rate. However, as mentioned above, the FAME yields reached a maximum value at KOH contents of 1.0 and 1.25 wt.%. From economic point of view, the optimal amount of KOH was therefore 1.0 wt.%.

4.3. Effect of molar ratio of methanol to oil

One of the most important variables affecting the yield of FAME is the molar ratio of methanol to oil. The stoichiometric molar ratio
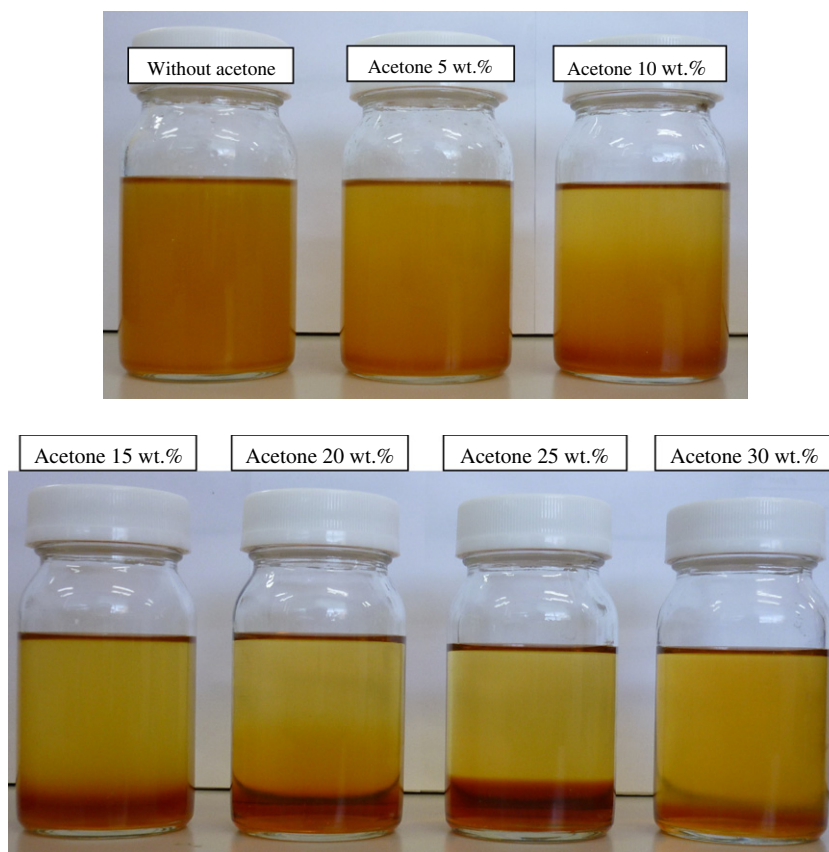
Photograph 1. GL separation at different amounts of acetone used after 30 min settling of the reaction products. The reaction conditions: molar ratio of methanol to WCO, 5:1; KOH catalyst, 0.5 wt.%; temperature, 25 ± 1 °C (*Notes*: the upper layer, FAME; the lower layer, GL).
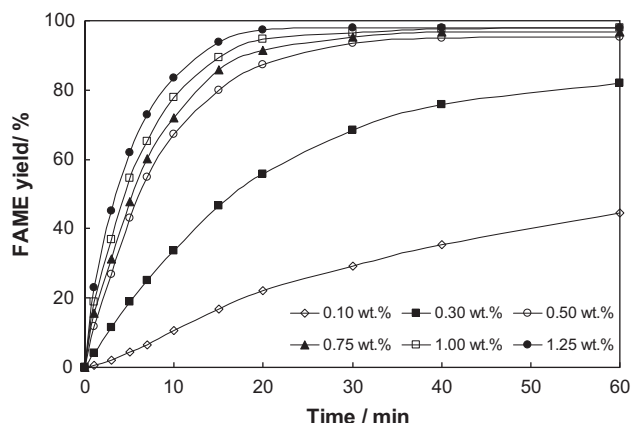
Fig. 2. Effect of the amount of KOH catalyst on the FAME yields. The reaction conditions: molar ratio of methanol to oil, 4.5:1; acetone to WCO, 25 wt.%; temperature, 25 ± 1 °C.

of methanol to oil for transesterification is 3:1, to transform 1 mol of GL and 3 mol of FAME. It is presumed that transesterification is an equilibrium reaction and therefore, excess methanol is desirable for the forward reaction. Several studies have suggested that the optimal molar ratio of methanol to oil for transesterification catalyzed by an alkaline catalyst is 6:1 [21,35–37]. For transesterification in supercritical methanol at 390 °C in the presence of hexane co-solvent, Sawangkeaw et al. reported that a 40.8:1 molar ratio of methanol to palm oil led to a FAME yield of 99.3% [38]. Therefore, to obtain higher yields of FAME, excess methanol should be used. In this study, molar ratios of methanol to oil from 3:1 to 5:1 were used with 1.0 wt.% KOH to determine the optimal molar ratio for transesterification.

Fig. 3 shows that in the initial stage of the reaction, up to 5 min, lower molar ratios increased FAME formation. The FAME yields beyond 5 min reaction time were 51.1%, 45.6%, 43.5%, 42.6% and 41.1% with molar ratios of methanol to oil of 3:1, 3.5:1, 4:1, 4.5:1 and 5:1, respectively. However, beyond 7 min reaction times, higher FAME yields were observed at higher molar ratios. At molar ratios of methanol to oil of 4.5:1 and 5:1, the conversion to FAME exceeded 98% after 30 min. When the molar ratio was decreased to the theoretical ratio of 3:1, the conversion to FAME decreased to 88%. This can be explained as follows. During the initial reaction stage, the concentrations of KOH catalyst in the methanol were 1.28, 1.10, 0.98, 0.85 and 0.77 mol L^{-1} for the experimental

conditions at methanol to oil molar ratios of 3:1, 3.5:1, 4:1, 4.5:1 and 5:1, respectively. This implies that the lower the molar ratios of methanol used, the higher was the concentration of KOH catalyst in the reaction mixture, because a constant 1.0 wt.% (against TG) KOH catalyst was used. The reaction therefore proceeded more rapidly and higher FAME yields were attained. This stage could be maintained until the formation of GL became significant, at which point GL began to act as a solvent for methanol and KOH, leading to the removal of a significant amount of reactants from the acetone reaction phase. This process may have caused slower FAME formation, so to maintain the reaction and increase the FAME yields in the latter stages of reaction, excess methanol would be preferable.

Interestingly, once GL formation became sufficiently large, more than 40 wt.%, the reaction rate slowed, which is consistent with the methanol and KOH being dissolved in the GL layer and thus removed from the reaction phase. Therefore, there is always competition between the reaction and dissolution of methanol into the GL layer. This phenomenon and the presence of reversible reactions are the two main reasons that transesterification of TG with alcohol does reach completion at a methanol to TG molar ratio of 3:1, the stoichiometric molar ratio. It is therefore confirmed that the retardation of FAME formation in the presence of by-product GL is not only due to the occurrence of the reverse reaction but also the removal of methanol and catalyst from the reaction phase. These findings are consistent with the discussion in Section 4.2. An increase in the amount of KOH catalyst can enhance the reaction rate, by making the consumption of methanol by reaction more favorable than its dissolution into the GL layer. Consequently, a molar ratio of 4.5:1 was optimal in terms of conversion to FAME is the present reaction system.

4.4. Effect of reaction temperature

Because vegetable oils and animal fats have high viscosity, methanolysis is usually performed near the boiling point of methanol, 65 °C to reduce the viscosity and enhance the mass transfer and the reactivity of the reactants. In the present study, the transesterification of WCO with methanol was performed within a limited range of temperatures (25–50 °C), which was lower than the boiling point of acetone, 56.53 °C.

As shown in Fig. 4, after a reaction time of 10 min, the FAME yields were 72, 83, 91 and 95% at 25, 30, 40 and 50 °C, respectively, illustrating the effect of temperature on the reaction. However, after a reaction time of 30 min, the FAME yields reached a constant
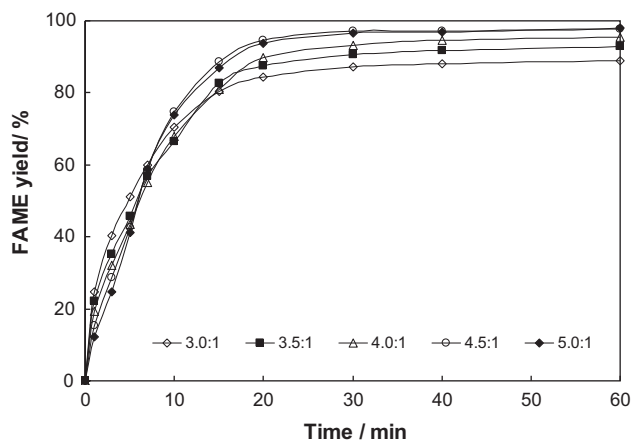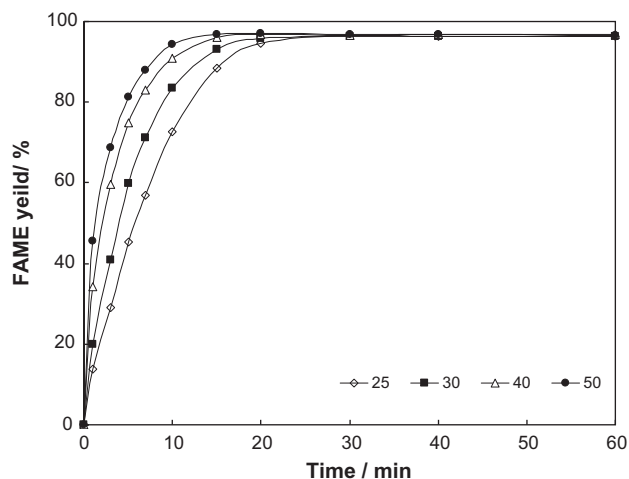

Fig. 3. Effect of the molar ratio of methanol to oil on the FAME yields. The reaction conditions: acetone to WCO, 25 wt.%; KOH catalyst, 1.0 wt.%; temperature, 25 ± 1 °C.

Fig. 4. Effect of temperature on the FAME yields. The reaction conditions: molar ratio of methanol to WCO, 4.5:1; acetone to WCO, 25 wt.%; KOH catalyst, 1.0 wt.%.
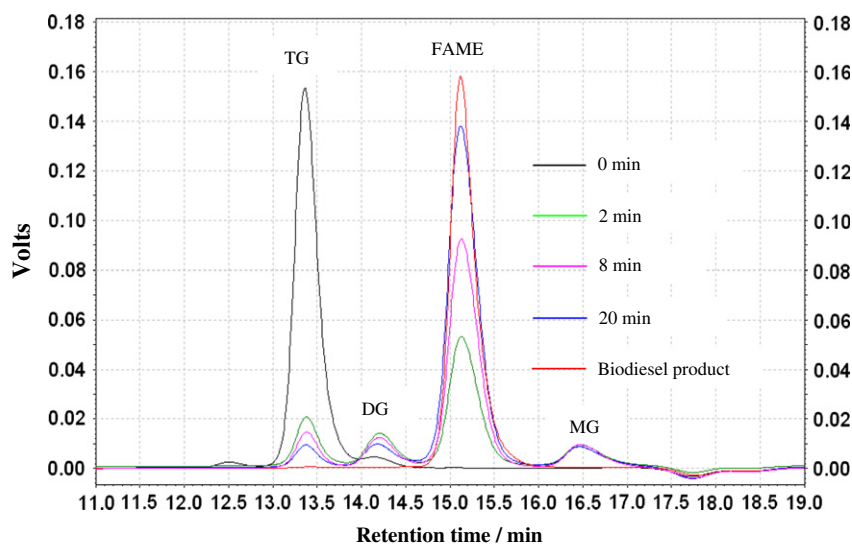
Fig. 5. HPLC chromatogram of reaction products versus time of WCO. The reaction conditions: acetone to oil, 25 wt.%; KOH catalyst, 1.0 wt.%; molar ratio of methanol to oil, 4.5:1, and temperature 25 ± 1 °C.

value of greater than 98% at all temperatures examined. This shows that temperature has a positive influence on the reaction rate but no significant effect on FAME yield. These results agree with those of Freedman et al. 1984, who reported identical FAME yields of 98, after 4 h reaction time at 60, 45 and 32 °C at a methanol to refined soybean oil molar ratio of 6:1 and a 1 wt.% NaOH catalyst [39]. In addition, an increase in temperature leads to acetone evaporation, higher energy consumption and soap formation. For these reasons, the optimum temperature for transesterification was considered to be 25 °C (ambient temperature).

4.5. Application of the co-solvent method to biodiesel production from a variety of oils

The optimal conditions were an acetone to oil content of 25 wt.%, 1.0 wt.% KOH, a methanol to oil molar ratio of 4.5:1 and a reaction temperature of 25 °C. The chemical properties of the other oils are similar to those of WCO, as given in Table 1. Therefore, the optimal conditions could be applied to produce biodiesel in the same manner as with WCO, with the exception that the reaction temperature for catfish oil was 35 °C because this oil solidifies below 25 °C. Fig. 5 shows the chemical compositions of the reaction products after various reaction times in the transesterification of WCO. The chemical compositions and phys-

ical properties of the products after transesterification of the oils and purification of crude BDF are given in Table 3. As shown in Table 3, the main quality factors of the BDF products in the present study met the Japanese Industrial Standard JIS K2390. Based on these results, we built a pilot plant with a capacity of 300 L per batch in Hanoi Vietnam, using this technique for the production of BDF (B100) from several types of oil in domestic use. Further research into the kinetics of transesterification using the co-solvent method is under way.

5. Conclusion

A transesterification process for the production of biodiesel from WCO assisted by an acetone co-solvent has been optimized. The optimal conditions are as follows: 25 wt.% acetone to oil; 1.0 wt.% KOH; methanol to oil molar ratio 4.5:1; and reaction temperature 25 °C. Under these conditions, the FAME yield is greater than 98%. This process significantly reduces the use of methanol compared to conventional methods (which use a methanol to oil molar ratio of 6:1). The results of this research were applied to produce BDF from several types of oil, and the main properties of the products satisfied the Japanese Industrial Standard (JIS K2390).

Table 3
Properties of biodiesel produced from canola, WCO and *Jatropha curcas* oils under the optimal conditions: acetone to oil, 25 wt.%; KOH catalyst, 1.0 wt.%; molar ratio of methanol to oil, 4.5:1; and reaction temperature 25 ± 1 °C.

Test parameter	Unit	Oils				JIS K2390[b]	
		Canola	WCO	Catfish[a]	*Jatropha*	Min.	Max.
Density (15 °C)	g cm^{-3}	0.875	0.887	0.883	0.872	0.860	0.900
Acid value	mg KOH g^{-1}	0.12	0.25	0.09	0.37	–	0.5
Methanol	wt.%	<0.01	<0.01	<0.01	<0.01	–	0.20
Total GL	wt.%	0.11	0.15	0.23	0.17	–	0.25
Free GL	wt.%	<0.01	<0.01	<0.01	<0.01	–	0.02
FAME	wt.%	98.30	98.11	97.02	97.91	96.5	
MG	wt.%	0.32	0.33	0.55	0.45	–	0.80
DG	wt.%	0.14	0.16	0.17	0.18	–	0.20
TG	wt.%	0.10	0.13	0.12	0.18	–	0.20

[a] Reaction temperature was 35 °C.
[b] JIS K2390: Japanese Industrial Standard for biodiesel (B100).

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