

MIXTURES OF VEGETABLE OIL AND USED FRYING OIL AS FEEDSTOCK FOR BIODIESEL PRODUCTION IN THAILAND

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Abstract

In this study, used frying oil was mixed with 3 fresh vegetable oils at various blending ratios (0.03-0.20 v/v) in order to study the feasibility of the biodiesel production from the mixed oil feedstocks by KOH-catalyzed methanolysis and also examine the effect of used frying oil content in oil feedstock on the reaction. The methanolysis was carried out with fresh vegetable oils (coconut, sunflower, and roselle oils), used frying oil (palm oil) and the mixtures of fresh vegetable oils and used frying oil in order to compare the methyl ester content in biodiesel products. The results revealed that biodiesels could be successfully produced from mixed oil feedstocks and the used frying oil- to-fresh vegetable oil ratio of 0.03 v/v was required in order to produced coconut-used frying oil and roselle-used frying oil biodiesels having methyl ester content higher than the minimum (96.5% w/w) prescribed by European Standard EN 14214 (2008) and Thai biodiesel (B100) specifications. The used frying oil content in oil feedstock had the negative effect on the reaction which resulted in the reduction of methyl ester content in biodiesel products. This was due to the free fatty acid and water in used frying oil caused soap formation which interfered with the reaction. The measured properties of biodiesels produced from mixed oil feedstocks at blending ratio of 0.03 v/v met with Thai biodiesel (B100) specifications, with the exception of the slight lower methyl ester content and lower viscosity of the sunflower-used frying oil biodiesel and the coconut-used frying oil biodiesel, respectively.

Keywords: Biodiesel, methanolysis, used frying oil, methyl ester content, used frying oil-to-fresh vegetable oil ratio

Introduction

Due to the depletion of world petroleum reserves, the rapidly increasing prices of petroleum, and the growing concern for the environment, the search for alternative fuels has currently attracted much attention (Meher *et al.*, 2006a; Balat and Balat, 2008). Among

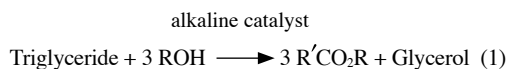
the alternative possible sources, biodiesel is becoming increasingly important as an alternative fuel in diesel substitution. It can be used in public transportation and performing farm engines (Tomasevic and Siler-Marinkovic, 2003). Biodiesel is monoalkyl esters of long

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chain fatty acids. It can be produced from vegetable oils and animal fats. These oils and fats have high viscosity (approximately 11-17 times higher than petroleum diesel) (Meher *et al.*, 2006a). Their high viscosity causes many problems in diesel engines when they are directly used as fuel. The conversion of these oils and fats to biodiesels leads to the reduction in viscosity to the acceptable level which results in the improvement of atomization, fuel vaporization, and combustion (Ramadhas *et al.*, 2005a). The characteristics of resulting biodiesel are close to those of petroleum diesel. Some important properties of biodiesel depend on the fatty acid composition of oil feedstock. Different types of oils have different types of fatty acids (Balat and Balat, 2008). The fatty acid composition of oil plays a significant role in the performance of biodiesel in diesel engines. Saturation fatty acid methyl esters improve stability and increase the cetane number and cloud point. On the other hand, more polyunsaturation reduces the stability, cetane number, and cloud point (Ramadhas *et al.*, 2005b).

The most common way of producing biodiesel is the alkali-catalyzed transesterification of vegetable oils and animal fats (Gerpen, 2005; Balat and Balat, 2008). The general equation for this reaction can be represented as follows:



The transesterification is also called alcoholysis. If methanol or ethanol is used as the reactant, it is called methanolysis or ethanolysis, respectively. Vegetable oils and animal fats are composed primarily of the triglycerides (Dorado *et al.*, 2004). In this reaction, triglycerides react with alcohol to produce alkyl esters and glycerol, the valuable by-product used in the soap, pharmaceutical, and cosmetic industries (Lewis, 1993). The most often used alcohol is methanol due to its low price and its physical and chemical advantages (Ma and Hanna, 1999). Sodium and potassium hydroxides are the commonly

used alkaline catalysts for this process (Gerpen, 2005; Ramadhas *et al.*, 2005a). It is known that the important factors that affect the alkali-catalyzed transesterification are the free fatty acid (FFA) and water contents (Freedman *et al.*, 1984; Ma and Hanna, 1999; Meher *et al.*, 2006a). To carry this reaction to completion, the oils having a FFA content less than 1% is required (Ghadge and Raheman, 2005; Tiwari *et al.*, 2007) and all materials should be substantially anhydrous (Meher *et al.*, 2006a).

Nowadays, various kinds of oils are used to produce biodiesel commercially such as palm, rapeseed, soybean, and sunflower oils. However, many published papers have reported that there are other oils which can be used for this purpose including jatropha, rice barn, karanja, cardoon, pumpkin seed, rubber seed, and used frying oils (Encinar *et al.*, 1999; Ramadhas *et al.*, 2005b; Leung and Guo, 2006; Meher *et al.*, 2006b; Krishnakumar *et al.*, 2008; Oliveira *et al.*, 2008; Schinas *et al.*, 2009). At present large quantities of used frying oils are produced from many places such as restaurants and households. These oils are reused several times and disposed of in inappropriate ways which cause health and environmental problems. One of several possibilities for utilization of these waste oils is to use them as feedstock for biodiesel production. This results in the alleviation of those problems. Actually, the waste used frying oil is one of the economical feedstocks for biodiesel production. However, it has different properties from the fresh refined or non-refined vegetable oils due to its higher impurities, FFA and water contents (Leung and Guo, 2006; Issariyakul *et al.*, 2008). Hence, it is not suitable for use as feedstock in the alkali-catalyzed transesterification due to the FFA and water causing the formation of soap which consumes the catalyst and reduces catalyst efficiency. The soap increases the viscosity of the reactant and the formation of gels, and interferes in the separation of glycerol from the methyl ester layer. These result in a low yield and low quality of biodiesel (Ma and Hanna, 1999; Meher *et al.*, 2006a; Issariyakul *et al.*, 2008). Although,

biodiesel can be produced from the oils having a high FFA content via acid-catalyzed transesterification, a longer time and acid-resistant reactor are required (Issariyakul *et al.*, 2008). To overcome this obstacle, the reduction of the FFA and water contents in used frying oil is necessary. One of the alternative ways to achieve this purpose is to blend it with the fresh vegetable oil having lower FFA and water contents. This is the way to make use of the used frying oil for the biodiesel production via a simple one-step alkali-catalyzed process. Recently, reports in the literature have shown that a satisfactory result was obtained when the biodiesel was produced from the mixture of canola oil and used cooking oil (Issariyakul *et al.*, 2008).

As a result, our research team was interested in the utilization of the mixture of fresh vegetable oil and used frying oil as feedstock for biodiesel production via alkali-catalyzed methanolysis. The fresh non-refined vegetable oils such as coconut, sunflower, and roselle oils were used. The objectives of this study are: (a) to study the feasibility of the mixtures of fresh vegetable oil and used frying oil as the feedstock for biodiesel production; (b) to investigate the effect of the used frying oil content in oil feedstock on the reaction.

Materials and Methods

Materials

The coconut, sunflower, and roselle oils were obtained from Sangsook Industry Company Limited (Bangkok, Thailand), the supermarket (Bangkok, Thailand), and Payakkaram Temple (Suphanburi Province, Thailand), respectively. The used frying oil (palm oil) was purchased from a local restaurant in Bangkok. The analytical reagent (AR) grade methanol, potassium hydroxide, and n-heptane were purchased from Ajax Finechem (New South Wales, Australia). The methyl esters such as methyl heptadecanoate, methyl esters of lauric, myristic, palmitic, stearic, oleic, linoleic, and linolenic acids were gas chromatography reference standard and

they were purchased from Fluka (Buchs, Switzerland).

Equipment

The reactions were carried out in a laboratory-scale setup. A 250 mL 3-necked flat-bottom flask was used as a reactor. Two of the 3 necks were equipped with a condenser and thermometer whereas the other was used as an inlet for the reactants. The reaction mixture was heated and well-stirred at 600 rpm (Vicente *et al.*, 2005) by a hot plate with a magnetic stirrer (Fisher scientific Model 11-302-50 SHP).

Experimental Conditions

The experiments were designed to study the feasibility of 3 mixtures of fresh non-refined vegetable oil and used frying oil as feedstock in the alkali-catalyzed methanolysis and the effect of the used frying oil content in oil feedstock on the reaction. For the latter purpose, the methanolysis of each fresh vegetable oil and used frying oil was also conducted to compare with the mixed oil feedstocks. In this study, potassium hydroxide (KOH) was used as a catalyst for all experiments and the other reaction conditions are shown in Table 2.

Alkali-catalyzed Methanolysis

The oil feedstocks, methanol, and potassium hydroxide were used in amounts established for each experiment. The oil was charged into the reactor and heated to the desired temperature. The solution of potassium hydroxide in methanol was prepared freshly in order to avoid the moisture absorbance and to maintain the catalytic activity (Meher *et al.*, 2006a, 2006b). The methanolic solution was then gradually added to the preheated oil. After the completion of the addition, the measurement of time was started. Heating and stirring were continued at atmospheric pressure. After the reaction, the product mixture was allowed to settle overnight under gravity in the separatory funnel. It was separated into 2 layers. The top layer consisted of methyl esters, residual unreacted methanol, catalyst, and impurities, whereas the bottom layer contained a mixture of glycerol, an excess of unreacted

methanol, catalyst, and impurities. The glycerol layer was drawn off and the methyl ester layer was then washed gently with hot distilled water at 60°C until the washing water had a pH value that was similar to that of distilled water. The pH of the washing water was measured by pH-indicator strips. This confirmed that the alkaline catalyst was completely removed. The wet methyl ester layer was then dried at 70°C by vacuum rotary evaporator (BUCHI Rotavapor Model R110, Flawil, Switzerland). Finally, the methyl ester content of each biodiesel product was determined by gas chromatography (GC).

Analytical Methods of Methyl Ester Content

Sunflower, Roselle, and used Frying Oils, the Mixture of Sunflower Oil and used Frying Oil, and the Mixture of Roselle Oil and used Frying Oil as Feedstocks

The methyl ester contents of biodiesels produced from these oils were determined by European Standard method EN 14103 (2003).

The Coconut Oil and the Mixture of Coconut Oil and used Frying Oil as Feedstocks

The methyl ester contents of biodiesels produced from these oil feedstocks were determined by GC technique. The gas chromatograph of Agilent 6890N that equipped with a flame ionization detector and a capillary column of crosslinked polyethylene glycol (HP-INNOWax, 25 m × 0.20 mm × 0.2 μm). The gas chromatograph oven was kept at 200°C for 5 min. The carrier gas was helium at a flow rate of 0.5 ml/min. The analysis was carried out by injecting 1 μL of sample solution into the gas chromatograph. The n-heptane was used as the solvent for preparing the sample solution. Each methyl ester in the sample was identified by comparing its retention time with the retention time of the standard of the methyl ester of FFAs. Methyl heptadecanoate was used as the internal standard for the methyl ester content determination.

Properties of Biodiesels

The biodiesels produced from the mixtures of fresh vegetable oil and used frying oil at the blending ratio of 0.03 v/v were sent to the Quality Control Division of PTT Public Company Limited to determine their properties by using standard methods. The results in comparison with those of Thai biodiesel (B100) specifications are shown in Table 3.

Results and Discussion

Characterization of Oil Feedstocks

In this study, the used frying oil was settled until the solid impurities were completely precipitated and the oil layer was then separated. All fresh vegetable oils and used frying oil were viscous and different in color. The sunflower and roselle oils were yellow whereas the coconut and used frying oils were brown and black in color, respectively. These oils were characterized by fatty acid composition, density, viscosity, FFA, and water contents. The results are presented in Table 1.

The results showed that the fresh vegetable oils and used frying oil had a higher viscosity than the average value of Thai petroleum diesel (3.068 mm²/s) by approximately 9-12 times. The used frying oil had higher FFA and water contents compared with those of fresh vegetable oils and the limit required for alkali-catalyzed transesterification. Hence, this used frying oil was not suitable to be the feedstock in the reaction.

Alkali-catalyzed Methanolysis

Effect of used frying oil content in oil feedstock

In this study, fresh vegetable oils, used frying oil and the mixtures of fresh vegetable oil and used frying oil were used as feedstocks for alkali-catalyzed methanolysis. The used frying oil-to-fresh vegetable oil ratios were 0.03, 0.05, 0.10, 0.15, and 0.20 v/v. The averages of the methyl ester contents in the biodiesel products are shown in Table 2.

It was found that the methyl ester contents reached the maximum values when the fresh vegetable oils were used as feedstocks. These results were in accordance with the results found by other researchers (Issariyakul *et al.*, 2008). On the other hand, used frying oil provided the lowest methyl ester content for all reaction conditions. The methyl ester content decreased with the increase in the used frying oil content in the oil feedstock. This similar trend was also obtained for all mixed oil feedstocks. In addition, it was observed that the light gelatinous precipitate (soap) and the emulsion between the biodiesel and washing water layers were occurred during the reaction and washing step, respectively, when the used frying oil and mixed oils were used as feedstocks. The amount of the gelatinous precipitate increased with further increased in the used frying oil content in oil feedstock. On the other hand, in the case of fresh vegetable oils, the light gelatinous precipitate and the emulsion formation were not observed during the reaction and washing step, respectively. These were due to the further increase of the used frying oil content which resulted in the increase of FFA and water contents in the oil feedstocks. The FFA and water caused the soap formation which interfered with the reaction as well as the separation of glycerol from the biodiesel product. It resulted in the low methyl ester content. Therefore, all these results indicated that the used frying oil content had a negative effect on the reaction and the FFA and water contents played a

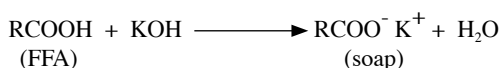
Table 1. Properties of oil feedstocks

Property	Method	Coconut oil	Sunflower oil	Roselle oil	Used frying oil
Fatty acid composition (%) ^a	AOAC (2005), 963.22, 969.33				
(i) Caprylic acid (C8:0)		3.4			
(ii) Capric acid (C10:0)		3.2			
(iii) Lauric acid (C12:0)		32.7			
(iv) Myristic acid (C14:0)		18.4			1.0 ^b
(v) Palmitic acid (C16:0)		13.1	6.2	18.2	42.8 ^b
(vi) Stearic acid (C18:0)		3.6	3.8	4.1	4.5 ^b
(vii) Oleic acid (C18:1)		12.9	38.6	33.3	40.5 ^b
(viii) Linoleic acid (C18:2)		4.4	50.1	38.2	10.1 ^b
(ix) Linolenic acid (C18:3)				2.1	0.2 ^b
Density at 15°C, kg/m ³	ASTM D 4052-96 (1996)	914.4	921.8	919.9	914.8
Kinematic viscosity at 40°C, mm ² /s	ASTM D 445-06 (2006)	27.52	32.68	36.35	38.40
Free fatty acid content, % w/w	AOAC (2000), 940.28	0.60	04.5	0.67	1.90
		(as lauric acid)	(as lauric acid)	(as lauric acid)	(as lauric acid)
Water content, % w/w	AOAC (1990), 984.20	0.0146	0.0840	0.0870	0.1077

^a Other fatty acids were present in amounts of < 1%.

^b Ma and Hanna, 1999

significant role in alkali-catalyzed methanolysis. The reaction of FFA and KOH to produce soap can be presented as follows:



In addition, the lower purity and yield of the biodiesel product might be due to some impurities in the used frying oil not being converted to methyl ester and still remaining in the ester layer. This result was also observed by some researchers (Leung and Guo, 2006).

It was found that the blending ratio of 0.03 v/v provided the highest methyl ester contents when compared with the rest of the blending ratios. At this ratio, the methyl ester contents of biodiesels produced from the mixture of coconut oil and used frying oil and the mixture of roselle oil and used frying oil were higher than the minimum (96.5%w/w) prescribed by European Standard EN

14214(2008) and Thai biodiesel (B100) specifications. Thus, this ratio can be applied for the large scale production using these mixed oil feedstocks. Although, the results showed that it was possible to produce biodiesel from the mixture of sunflower oil and used frying oil, further investigation of the optimum conditions which provide the methyl ester content higher than 96.5% w/w is necessary.

Although, the prior research papers reported that the alkali-catalyzed transesterification of used frying oil with high FFA and water contents could be achieved but the higher operating conditions were required (Gerpen, 2005; Leung and Guo, 2006) or the FFA and water contents had to reduce to the acceptable level through pre-treatment process (Canakci and Genpen, 2001; Leung and Guo, 2006; Wang *et al.*, 2007). These lead to the increasing of the biodiesel production cost. In addition, the alternative way for biodiesel production from used frying oil is the acid-

Table 2. The variation of methyl ester content with used frying oil content in oil feedstocks

Feedstock ^a	Methyl ester content (%w/w)	Feedstock ^b	Methyl ester content (%w/w)	Feedstock ^c	Methyl ester content (%w/w)
Coconut	98.4	Sunflower	96.9	Roselle	99.4
Mixed oil used frying oil/vegetable oil ratio (v/v)		Mixed oil used frying oil/vegetable oil ratio (v/v)		Mixed oil used frying oil/vegetable oil ratio (v/v)	
0.03	97.5	0.03	95.4	0.03	97.2
0.05	95.3	0.05	93.7	0.05	95.5
0.10	92.5	0.10	91.3	0.10	92.6
0.15	89.7	0.15	89.6	0.15	91.5
0.20	87.1	0.20	87.8	0.20	89.0
Used frying oil	67.3	Used frying oil	65.5	Used frying oil	68.7

^a Reaction condition: Methanol:oil of 0.4 v/v, catalyst concentration of 1.5% w/v of oil, 60°C, and 60 minutes

^b Reaction condition: Methanol:oil of 8:1, catalyst concentration of 1.0% w/w of oil, 60°C, and 90 minutes (Vicente *et al.*, 2005)

^c Reaction condition: Methanol:oil of 8:1, catalyst concentration of 1.5% w/w of oil, 60°C, and 60 minutes

catalyzed transesterification which can be used to prevent the soap formation (Canakci and Genpen, 1999; Zheng *et al.*, 2006). However, this process required the longer reaction time and the acid-resistible reactor (Issariyakul *et al.*, 2008). Therefore, in this study, the utilization of the mixture of fresh vegetable oil and used frying oil as feedstock is an alternative way to make use of the used frying oil for the biodiesel production through a simple one-step and low-cost alkali-catalyzed process. Although, in this study, the ratio of 0.03 v/v seems to consume only a little amount of used frying oil, in the industrial biodiesel production, this ratio leads to a large scale consumption of used frying oil.

Properties of Biodiesels Produced from the Mixtures of Fresh Vegetable Oil and Used Frying Oil

Table 3 shows that the measured properties of the 3 biodiesels were within the

limits prescribed by Thai specifications, with the exception of the slight lower methyl ester content and lower viscosity of the sunflower-used frying oil biodiesel and the coconut-used frying oil biodiesel, respectively. However, the viscosity of coconut-used frying oil biodiesel was very close to that of Thai petroleum diesel and it was within the limits of ASTM D 6751-07b (2007) (1.9-6.0 mm²/s). This was because the coconut triglyceride consisted of 57.5% short chain fatty acids comprised primarily of lauric acid whereas the sunflower and roselle triglycerides consisted chiefly of long chain fatty acids such as oleic and linoleic acids. Thus, biodiesel obtained from the mixture of coconut oil and used frying oil contained a high proportion of low molecular weight methyl esters, especially methyl laurate. On the other hand, the other 2 biodiesels contained mainly methyl oleate and methyl linoleate (high molecular weight methyl esters). This resulted in the lower viscosity of biodiesel produced from the mixture of

Table 3. Measured properties of biodiesel products in comparison with Thai biodiesel (B100) specifications

Property	Method	Limit	Biodiesel		
			Coconut-used frying oil	Sunflower-used frying oil	Roselle-used frying oil
Methyl ester content, % w/w	EN 14103 (2003)	96.5	97.5	95.4	97.2
Density at 15°C, kg/m ³	ASTM D 4052-96 (1996)	860-900	879.4	883.9	886.2
Kinematic viscosity at 40°C, mm ² /s	ASTM D 445-06 (2006)	3.5-5	3.057	4.286	4.549
Flash point, °C	ASTM D 93-02a (2002)	120 min	> 120	> 120	> 120
Copper strip corrosion (3 h at 50°C)	ASTM D 130-94 (1994)	No. 1 max	1a	1a	1a
Water content, mg/kg ISO 12937 (2000)	ISO 12937 (2000)	500 max	420	440	390
Total acid number, mg KOH/g	ASTM D 664-01 (2001)	0.5 max	0.28	0.15	0.31
Linolenic methyl ester content, % w/w	EN 14103 (2003)	12.0 max	0	0	2.088

coconut oil and used frying oil as compared with the other 2 biodiesels.

Conclusions

This study demonstrated that the mixtures of fresh vegetable oil (coconut or sunflower or roselle oil) and used frying oil could be used as feedstock for biodiesel production via alkali-catalyzed methanolysis. The used frying oil content in oil feedstocks had a negative effect on the reaction which was due to its higher FFA and water contents. The blending ratio of 0.03 v/v provided the best results, especially for the biodiesels produced from the mixtures of coconut-used frying oil and roselle-used frying oil. The measured properties of biodiesels met with Thai biodiesel (B100) specifications, with the exception of the slight lower methyl ester content and lower viscosity of sunflower-used frying oil biodiesel and coconut-used frying oil biodiesel, respectively. Therefore, these mixtures of fresh vegetable oil and used frying oil can be recommended as a supplementary oil feedstock for biodiesel production in Thailand if engine performance tests provide satisfactory results.

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