



A Two-step Catalytic Production of Biodiesel from Waste Cooking Oil

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ABSTRACT

Waste cooking oil (WCO) was used as a potential feedstock for biodiesel production. High level of free fatty acids (9.85% w/w) in WCO makes it an undesirable substrate for direct transesterification reaction. To solve this issue, a two-step process was implemented in this research. Firstly, esterification reaction was performed in the presence of sulfuric acid as a common acid catalyst to reduce the amount of free fatty acids (FFA) to less than 1.5% w/w. The triglycerides (TGs) in WCO were transesterified with methanol catalyzed by potassium hydroxide in the second step of reaction. Several effective parameters were evaluated in terms of reaction temperature (323-363 K), reaction time (0.5-3 h) and catalyst concentration (0-5.52% w/w). The optimum reaction temperature, reaction time and catalyst concentration were 355 K, 3 h and 3.68~4% w/w, respectively. In fact, maximum FFA conversion of 87 wt% was obtained. The biodiesel synthesis condition in the second stage of reaction was defined as the reaction temperature (338 K), methanol/ WCO molar ratio (6:1), KOH concentration (1% w/w) and reaction time (90 min). Under the above conditions, maximum TG conversion of 96.66 wt% was obtained.

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1. INTRODUCTION

Recent developments in life style and significant growth of population have gradually increased fossil fuel consumptions. Excessive demand for non-renewable fuels implies depletion of fossil fuel reserves, rising prices and increasing environmental concerns due to rising greenhouse gas emissions such as carbon monoxide, carbon dioxide, sulphur oxides, heavy metals, polycyclic aromatic hydrocarbons and volatile organic compounds. Several studies have been carried out in order to find new renewable and sustainable energy sources to substitute the fossil fuel. Among the various types of alternative fuels produced from renewable resources, biodiesel is considered as a promising alternative substitute for petroleum derivatives fuels [1-4].

Three general approaches are recommended for production of biodiesel including micro-emulsion, transesterification and pyrolysis. The most commonly used method for converting oils to biodiesel is through the transesterification reaction. Transesterification is

defined as a reaction between vegetable oil or animal fat with an alcohol in the presence of a base or an acid catalyst to produce monoglycerides and diglycerides as intermediate products and esters (biodiesel) and glycerol as final products [5].

The methyl esters produced by transesterification of vegetable oil have low viscosity and improved combustion properties compared to those of fossil fuels and crude vegetable oil [6, 7]. In addition, the obtained glycerol can be used as byproduct in developed applications in terms of animal feed, carbon feedstock in fermentations, polymers, surfactants, intermediates and lubricants [8].

In spite of all advantages of biodiesel; one drawback in biodiesel production is high production cost; which is related to the high price of the vegetable oils. Applying non-edible or waste cooking oils because of their low cost and the environmental advantage of the residue disposal can lower the raw materials cost up to about 60-70%.

“Waste Cooking Oil” (WCO) is vegetable oil which has been used in food production and usually contains free fatty acids (FFA), water and other impurities which determine the quality of biodiesel. WCO can be

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supplied from many different sources including domestic (cooking in kitchen), commercial (cooking in restaurant or fast food centers) and industrial (factories that produce food) sources. Their main use is in the production of animal feed and in the manufacture of soaps and biodegradable lubricants [9, 10].

Several potentially useful catalysts have been studied in transesterification reaction which significantly increased the reaction rate in comparison to the reaction without catalyst. Without a catalyst, high reaction yield can be obtained only under extreme conditions of temperature, pressure and time which lead to adverse effects such as isomerization, polymerization and splits. Catalysts are classified into two categories: homogeneous (alkaline - NaOH, KOH acid - H₂SO₄ and alkali-alcoholic MeOH) and heterogeneous (CaO, MgO, BaO and ZnO/Al₂O₃, oxides of alkali metal-based zeolite, ion exchange resins) [11].

Transesterification reaction can be carried out in the presence of homogeneous alkaline catalysts even at relatively low temperature and pressure. The main disadvantage of them is their weak performance in the presence of high levels of FFA and moisture in oilseeds which leads to saponification and low ester yield. The homogeneous acid catalyst is preferred when feedstock contains high FFA content. Acid-catalyzed reaction is much slower and needs higher temperature (373 K - 473 K), pressure and molar ratio of methanol to oil (1:10 to 35) than alkali-catalyzed one. Moreover, corrosion of equipments and environment contamination are drawbacks associated with use of homogeneous catalysts [12].

Generally, WCO contains a large percentage of FFA. As a result of utilizing alkaline catalysts; soaps are produced in reaction vessel. To avoid saponification, numbers of pretreatment methods are proposed, including steam distillation, extraction by alcohol, and esterification by acid catalysis. However, the common pretreatment is esterification of the FFA with methanol in the presence of acidic catalysts [9, 13, 14].

In this paper, an acid catalyst was employed to carry out esterification reaction in the first step and in the second step; alkaline catalyst transesterification was performed with product of previous step as the reactant. WCO was used as a worthless and abundant feedstock to produce biodiesel. The effect of several operation parameters on conversion of TG was considered and optimum conditions were obtained.

2. MATERIALS AND METHODS

2.1. Materials WCO was collected from Babol Noushirvani University canteen. Sulfuric acid (H₂SO₄) 98% laboratory grade and potassium hydroxide (KOH)

85% were purchased from Merck (Darmstadt, Germany).

2.2. Pretreatment of the Waste Cooking Oil

Solid particles, salt, pepper and spices in WCO were removed by filtration. The WCO sample containing water and other impurities was centrifuged at 15000 rpm. Water content of WCO was dried over Na₂SO₄ crystals for 72 h. Then, the WCO was mixed with *n*-hexane (1:3 oil/ hexane, volume ratio) to remove the remaining impurities. After 72 h of drying period, the oil was separated from *n*-hexane in a rotary evaporator. In order to make sure about complete removal of *n*-hexane, the residue was placed in freeze dryer. The composition of FFA in WCO was determined by following method:

5 grams of the filtered WCO was poured into a 250 ml Erlenmeyer flask, and then 20-30 ml of neutralized anhydrous ethanol was added. Stirring was continued until the oil was completely dissolved in the solvent mixture. A 25 ml burette was filled with about 10-15 ml of ethanolic KOH solution (by solving potassium hydroxide in ethanol) and 5-6 drops of phenolphthalein (1% in ethanol) and the KOH solution was added to the mixture in Erlenmeyer drop by drop. When the color of the mixture changed, the mixture was left for 30 second to be sure of sustainability of the color. Equation (1) was used to determine the concentration of FFA in WCO:

$$\% \text{ FFA} = \frac{\text{avg.mol.wt} \times N \times 100 \times V}{1000 \times W} \quad (1)$$

In the above equation N is normality of KOH, V is volume of KOH used for titration, avg.mol.wt is average molecular weight of oil (278.22 g / mol) and W is initial weight in grams.

The fatty acid components of WCO were determined by GC-MS. Three saturated fatty acids (C (16:0), C (18:0), C (20:0)) and four unsaturated fatty acids (C (16:1), C (18:1), C (18:2), C (18:3)) were detected in WCO sample. The compositions of the fatty acids are summarized in Table 1.

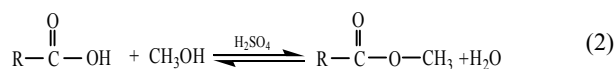
TABLE 1. Composition of the fatty acids in WCO

Fatty Acid	(wt %)
C 14: 0	0
C 16: 0	28.91
C 16: 1	1.45
C 18: 0	0.93
C 18: 1	26.51
C 18: 2	27.44
C 18: 3	4.6
C 20:0	0.33
FFA content	9.85
Water content	0.03

2. 3. Homogenous Acid-catalyzed Transesterification

A 100 g of the filtered WCO containing 9.85 wt% FFA was poured in a three necked round-bottom flask equipped with a reflux condenser and a temperature controller and a stopper. The flask was immersed in a constant-temperature bath while stirring rate was fixed at 600 rpm to avoid mass transfer limitations through the process. A solution of sulfuric acid in methanol was prepared by slowly introducing H_2SO_4 into methanol. Esterification reaction was carried out with methanol to TG molar ratio of of 8:1 and 4 levels of reaction times, 0.5, 1, 2 and 3 h. The effect of temperature on the conversion of FFA was studied at different temperatures from 323 to 363 K while amount of H_2SO_4 as catalyst was varied from 0 to 3 ml [15-17].

The prepared solution was added into the pretreated WCO and stirred. The resulting product mixture was centrifuged. Upon the centrifugation at 15000 rpm, two phases were formed, the upper layer (the oily phase, consisting of oil, methyl ester, un-reacted oil and some impurities) and a bottom phase (the waste phase, consisting water and sulfuric acid). The upper layer was separated, washed with dionized water and dried over Na_2SO_4 for 48 h and was kept to produce biodiesel by transesterification in the second step. Equation (2) demonstrates the mechanism of acid- catalyzed process for biodiesel synthesis:



In this reaction, triglycerides (esters of long chain carboxylic acids) are mixed with methanol. Presence of the carbonyl group in triglyceride structure and its protonation forms carbonation through the reaction. Addition of alcohol may cause a nucleophilic attack and gives an intermediate which can lose a proton to form a new ester. Since the reaction between carbonation and water can produce carboxylic acids, it has been proposed that this reaction should be performed in the absence of water [9, 18].

2. 4. Homogenous Base-catalyzed Transesterification

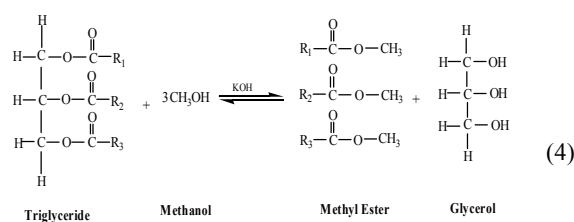
The acid-catalyzed oil was placed in the reaction vessel and heated to achieve the temperature of 338 K. The initial solution was prepared by adding 1g of KOH in 90 ml of methanol. This solution was added into the pretreated oil and stirred at 338 K for 90 min (however the reaction time is between 1 to 4 h commonly, but more than 80% of the reaction is completed in the first 45 min). After completion of the reaction, the mixture was centrifuged. Upon the centrifugation at 15000 rpm, two phases were formed; the upper layer was biodiesel and was separated from lower glycerin layer. The biodiesel layer was washed 3-5 times with dionized water to remove soap and excess KOH.

Finally, the product was dried over Na_2SO_4 and the yield was calculated using the following equation:

$$\text{Yield (\%)} = \left(\frac{\text{Wt. of biodiesel}}{\text{Wt. of initial oil}} \right) \times 100 \quad (3)$$

Often some excess methanol was present in the reaction which was recovered after completion of the reaction by slightly heating ($64^\circ C$) in a rotary evaporator.

The products were analyzed using GC-MS spectrometer (the MS used was an Agilent 5973 equipped with an Agilent 6890N GC) equipped with a capillary column (HP-5). Wiley libraries were used as reference databases. Equation (4) shows mechanism of the base-catalyzed transesterification for biodiesel production:



R_1, R_2 and R_3 = hydrocarbon chain from 13 to 21 carbon atoms of the fatty acid

In base-catalyzed transesterification reaction, at first a base reacts with an alcohol to produce an alkoxide and a protonated catalyst. The alkoxide attacks the carbonyl group to create a tetrahedral intermediate, from which the alkyl ester and the anionic diglyceride are formed. In the latter step, the catalyst loses its proton thus, regeneration of the active species may occur. The regenerated catalyst is now usable for next catalytic cycle. Diglycerides and monoglyceride are converted to glycerol and ester through the same mechanism as described above [12, 15]. The schematic process flow diagram for the two-step biodiesel production from WCO including pretreatments of WCO are shown in Figure 1.

3. RESULTS AND DISCUSSIONS

3. 1. Sulfuric Acid-catalyzed Process for Biodiesel Production

3. 1. 1. Effect of Reaction Time on Conversion

Since the base-catalyzed transesterification is very sensitive to the purity of the reactants; different parameters were optimized in the acidic esterification reaction in order to reduce FFA content and simplicity of transesterification reaction [19]. Effect of reaction time on the FFA conversion is shown in Figure 2. It was found that the conversion of FFA to FAME (fatty acid methyl ester) proceeded with reaction time. It has been reported that, if the oil contains high amounts of FFA

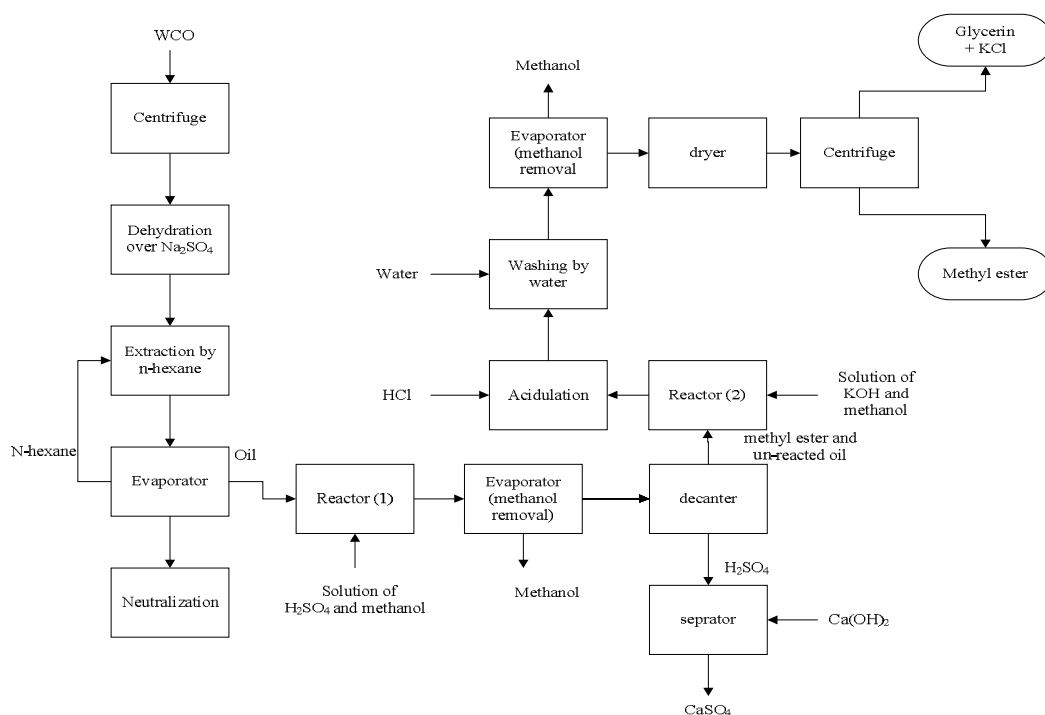


Figure 1. Schematic process flow diagram for the two-step biodiesel production from WCO

(>1% w/w), FFAs react with base catalysts and form soap [20]. So, the FFA content of the sample was reduced from 9.85% to about 1.5% after acid catalysis esterification. After 30 min from the starting reaction, still 5.4% of FFA content was remained in WCO sample because sufficient contact time was not provided. Whereas FFA content at the end of the second hour of reaction was less than 1.5%. Therefore, 3 h of reaction time was sufficient for the completion of the esterification reaction (most of FFA were converted into biodiesel), and final conversion of 87% was obtained. The reaction conversion did not significantly improve even when long reaction time was given.

3. 1. 3. Effects of Catalyst (H_2SO_4) Concentrations

Catalyst concentration was optimized in this study because both low and high amount of catalysts reduce the efficiency of biodiesel production; especially when a strong acid such as sulfuric acid is used. The relationship between H_2SO_4 concentration and conversion of esterification reaction is presented in Figure 4. As shown in this figure; conversion of the esterification in the absence of catalyst was 23%. When 1 wt% of catalyst was added, 75% of FFA was converted into FAME in 3 h. However, when the amount of catalyst exceeded 2.5 wt%, the rate of

reaction did not improve; because equilibrium was established in the reaction.

Thus, the optimal conditions obtained in sulfuric acid-catalyzed process were as follows; the reaction time (3 h), amount of catalyst (3.68 wt%) and methanol to TG molar ratio 8:1. The conversion of FFA was 87% when the reaction was performed under these conditions. Table 2 summarized the obtained results of all experiments.

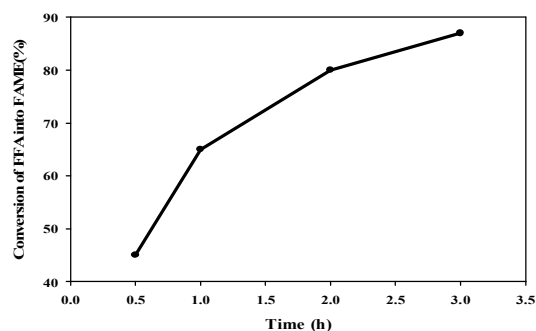
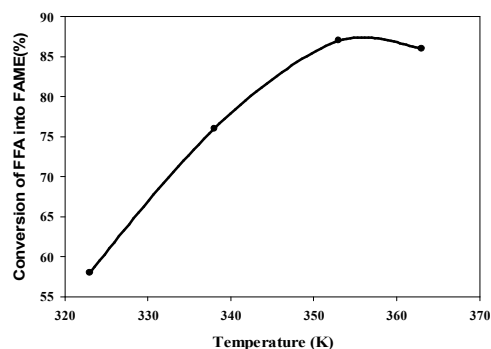
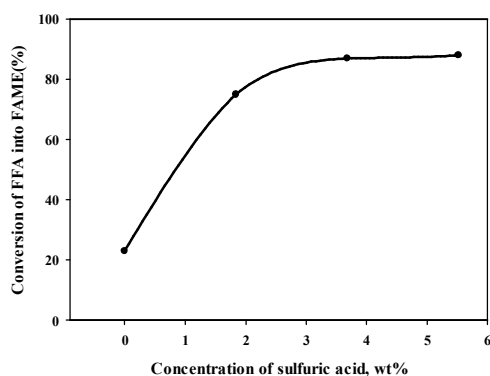


Figure 2. Influence of reaction time on the conversion of FFA (molar ratio of methanol to TG 8:1, reaction temperature of 355 K and 3.68% w/w of sulfuric acid)

TABLE 2. FFA content and conversion after acid-catalyzed reaction

Run No.	Reaction Temperature (K)	Reaction Time (h)	Amount of Catalyst (wt %)	Methanol: TG in Mole Ratio	FFA Content (wt %)	Conversion (%)
1	323	3	3.68	8:1	4.137	58.33
2	338	3	3.68	8:1	2.34	76.21
3	353	3	3.68	8:1	1.35	86.28
4	363	3	3.68	8:1	1.46	85.14
5	355	0.5	3.68	8:1	5.42	44.89
6	355	1	3.68	8:1	3.40	65.42
7	355	2	3.68	8:1	2.00	79.68
8	355	3	3.68	8:1	1.25	87.21
9	355	3	0	8:1	7.57	23.13
10	355	3	1.84	8:1	2.48	74.76
11	355	3	5.52	8:1	1.18	87.95

**Figure 3.** Influence of reaction temperature on the conversion of FFA (molar ratio of methanol to TG 8:1 and 3.68% w/w of sulfuric acid)**Figure 4.** Effect of concentration of sulfuric acid (wt%) on the conversion of FFA (molar ratio of methanol to TG 8:1 and the reaction temperature of 355 K)

3. 2. Potassium Hydroxide-catalyzed Process for Biodiesel Production The first step of this study was carried out to reduce the FFA content of WCO by

an acid-catalyzed esterification process. Different parameters were evaluated to optimize the FFA content for further processing. In the second stage, pre-treated WCO was considered as the reactant for the transesterification process; where TG is converted to FAME using an alkaline catalyst. The pretreated WCO under optimum conditions were further transesterified into biodiesel using an alkaline catalyst. The optimum conditions were molar ratio of methanol to WCO (6:1), reaction temperature (338 K), reaction time (90 min), stirring rate of 600 rpm and the amount of KOH as alkaline catalyst (1% w/w). Under these conditions, final conversion of 96.66% was obtained.

The compositions of biodiesel produced from WCO are summarized in Table 3.

According to Table 3, the biodiesel produced from WCO by two-step transesterification reaction was lighter and had lower density than biodiesel synthesized from other oils and methods [23, 24].

TABLE 3. Analysis of biodiesel produced from WCO

Biodiesel Components	Linear Formula	(%)
Hexadecanoic acid, methyl ester, Methyl palmitate	$C_{17}H_{34}O_2$	30.85
9,12-Octadecadienoic acid (Z,Z), methyl ester	$C_{19}H_{34}O_2$	29.91
8-Octadecenoic acid, methyl ester, Methyl oleate	$C_{19}H_{36}O_2$	16.96
Methyl dihydromalvalate	$C_{19}H_{36}O_2$	7.71
9-Octadecenoic acid (Z), methyl ester	$C_{19}H_{36}O_2$	9.32
α -Linolenic acid, methyl ester, Methyl alpha-linolenate	$C_{19}H_{32}O_2$	4.38
4,4-Dimethyl-1,1:3,1-terphenyl -2-carboxylic Acid, methyl ester	$C_{22}H_{20}O_2$	0.87

4. CONCLUSION

The results of present study expressed that WCO, despite having high FFA content could be a potential substitute for the high cost crude oil. A pre-treatment stage was performed to remove impurities including waste water, salt and other impurities from WCO. Then FFA content was reduced using sulfuric acid as a catalyst in the process of esterification, and finally TG was converted to biodiesel in potassium hydroxide-catalyzed transesterification reaction. The experiments were performed by varying several parameters in order to optimize the reaction conditions. Esterification reaction was carried out in 3 h with 3 ml of acid per 100 g of oil at 355 K. The remained WCO with less than 1.5 wt% of FFA content was used for the transesterification reaction with following conditions: reaction temperature of 338 K, reaction time of 90 min and potassium hydroxide concentration of 1% w/w. Finally, 96.66% of WCO was converted to biodiesel.

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ضایعات روغن پخت و پز به عنوان ماده اولیه برای تولید بیودیزل استفاده گردید. محتوی اسید چرب موجود در این ضایعات (۹/۸۵٪ وزنی) موجب می شود که این روغن برای واکنش ترانس استریفیکاسیون مستقیم مطلوب نباشد. برای حل این مشکل، یک فرآیند دو مرحله ای پیشنهاد شد: در مرحله اول استریفیکاسیون در حضور یک کاتالیست اسیدی متداول مانند اسید سولفوریک انجام شد. در مرحله دوم، تری گلیسریدهای روغن زاید با متانول در حضور کاتالیست پتاسیم هیدروکسید وارد واکنش شدند. واکنش کاهش اسید چرب آزاد در مرحله اول در رنج دمایی ۳۲۳-۳۶۳ کلوین در مدت زمان واکنش ۰/۵ تا ۳ ساعت و غلظت اسید در آن به عنوان کاتالیست از ۰ تا ۵/۵۲ انجام شد بهترین شرایط واکنش در دمای ۳۵۵ درجه کلوین در مدت زمانی ۳ ساعت که در آن غلظت اسید سولفوریک ۳/۶۸ ~ ۴٪ بود بدست آمد که بیشترین تبدیل اسید چرب آزاد در این مرحله ۸۷٪ وزنی بوده است. شرایط سنتز بیودیزل در مرحله دوم در دمای ۳۳۸ درجه کلوین، نسبت مولی متانول به روغن ۱:۶ در زمان ۹۰ دقیقه که مقدار باز در آن ۱٪ وزنی بوده، انتخاب شده است. در این شرایط بیشترین تبدیل تری گلیسرید ۹۶/۶۶٪ وزنی بوده است.

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