Temperature Assessment and Process Optimization of Alkali Catalyzed Transesterification of Waste Cooking Oil Using Microwave Flow System

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Abstract

The utilization of non-edible/waste oils and microwave heating system are expected to reduce the cost of biodiesel and contribute to reducing global food demand. In this study, the continuous biodiesel production from waste cooking oil (WCO) by the use of a microwave flow system has been investigated. The utilization of WCO as a biodiesel feedstock is expected to minimize the industrial biodiesel production. A household microwave was modified as a biodiesel reactor for continuous transesterification. The effect of transesterification reaction parameters such as methanol/WCO molar ratio (3:1, 6:1 and 9:1), quantity of the catalyst (0.5%, 0.75%, 1% and 1.5% in weight of KOH) and flow rate of reactant (6, 10.5 and 14.5ml/min) in various microwave output power (360, 450, 540, 630 and 720 watts) on the biodiesel yield has been evaluated. Results showed that WCO can be converted to biodiesel (96.53%) within 4min under methanol/oil molar ratio of 6:1, 0.75 wt. % catalyst, 6ml/min flow rate in 450W microwave output power. Moreover, the results explained that the high yield can be obtained when the reaction temperature is in the range of 40-45 °C. Microwave assisted continuous transesterification process is simple, practical, time-saving and no requirement to pretreatment of feedstock, offering alternative production method to produce biodiesel according to European Union draft standards.

Keywords: Biodiesel; Waste cooking oil (WCO); Transesterification; Microwave heating; Flow process

Introduction

The consumption of petroleum fuels as an important energy in transportation and industrial sectors, cause to increase greenhouse gas emission and environmental problems. Moreover, unavoidably increases petroleum fuels price and limitation of petroleum reserve assure the research for the development of alternative bio-fuels from renewable resources [1]. It known that Rudolph Diesel was firstly examined peanut oil in the internal combustion engine. However, some problems such as coling in the cylinder and at the fuel injector, ring sticking, gum for motion and lubricant thickening prevented the use of edible oils in the engines. Therefore, the researchers suggested conversion of vegetable oils to esters of short chain alcohols (biodiesel) to alleviate all the above problems. Thus, biodiesel has been widely accepted for biodiesel production instead of edible vegetable oils to do not be rival the global foods demand. Waste cooking oil (WCO)is an expectable feedstock for biodiesel production which generated everyday from restaurants and food processing industries [5,6]. Conversion of WCO to biodiesel helps to solve of environmental and transportation problems and also reduces the cost of biodiesel [7,8].

Biodiesel are generally produced by the conventional heating system which appears to be inefficient due to many loss of energy to the ambient and longer reaction time [9]. Therefore, alternative heating system has been used to reduce reaction time and energy consumption. The super and subcritical alcohol (usually methanol) [10-12], Ultrasonic [13,14], and microwave processes [15,16], were assessed in transesterification reaction. Although the super-critical process reduces the reaction time to 2-4min, very expensive reactors and high energy require due to operating of process at high pressure and temperature [17]. The ultrasonic process also is an appropriate alternative heating system for biodiesel production in short time, but the process requires high amount of co-solvent and alcohol [18]. It seems that the microwave irradiation process...
is a suitable heating system for biodiesel production with energy-efficient and a quick process because of the transfer of heat directly to the reactants [19,20].

Microwave-assisted transesterification can be carried out by batch or continuous process. Many researches were performed on biodiesel production from oils by batch process [21-24]. Although, the batch transesterification process is simple and presents several technical advantages, there is much inefficiency associated with batch production. One of the biggest problems with batch production is waste a great deal of time due to stopping the machines and equipment and then cleaned and prepared to use again. Another problem is the need for large size of vessel [25,26]. However, reaction vessels cannot be very large for biodiesel production by microwave irradiation due to limitation of penetration of microwave irradiation and safety [24,27]. The significant quantities of reagents can be processed in the continuous flow and only a relatively small quantity of material is in the microwave field at any one time [28,29]. Several reports described the use of domestic microwave ovens as continuous-flow process for biodiesel production from soybean oil [29], jatropha oil [30], rapeseed oil [31], and waste frying palm oil [9].

Biodiesel production through transesterification reaction typically depends on the reaction temperature, reaction time, catalyst concentration and alcohol to oil ratio [7,29]. The reaction temperature in continuous microwave assisted biodiesel production depends on methanol to oil ratio, flow rate of reactants and microwave output power and the reaction time depends on flow rate of reactants [32]. Therefore, the aim of this study is assessment and optimization of influence parameters on continuous microwave assisted biodiesel production from WCO by KOH as homogeneous catalyst. In addition, change of the reaction temperature during transesterification reaction and the effect of these parameters on the reaction temperature were investigated.

**Material and Methods**

**Materials**

The WCO was prepared from central kitchen of Ferdowsi University of Mashhad. WCO was filtered two times for removing the food particles and heated for 1h at 120 °C to remove moisture. The properties of WCO were listed in Table 1. Methanol and potassium hydroxide were purchased from Merck co.

| Property                           | Value |
|------------------------------------|-------|
| Density to 25 °C (Kg. m⁻³)         | 880   |
| Water content (%)                  | 2.75  |
| Acid value (mg KOH. g sample⁻¹)    | 2.75  |
| Saponification value (mg KOH. g sample⁻¹) | 185.8 |
| Iodine value (g I₂. 100 g sample⁻¹) | 80    |

**Experimental system**

The conversion of WCO was performed by continuous microwave assisted transesterification reaction. This system consists of silicone tube of 4mm ID, length of 4m, which was coiled into the microwave oven and connected to the inlet mixture tank and the outlet reservoir. Moreover, the temperature of feedstock and product was systematically predicted by resistance temperature detector (Pt100) which connected to a digital thermometer for monitoring the reaction temperature. The feedstock was pumped by a peristaltic pump to microwave oven.

This oven is a domestic microwave (Daewoo, KOC9N2TB, 2.45GHz and 900W output power) which was modified with two holes for inlet and outlet. First of all, the dissolved potassium hydroxide in desirable methanol was prepared basis on the methanol/WCO molar ratio (6, 9, 12 and 15) and on the required amount catalyst (0.5, 0.75, 1 and 1.5wt.%). Then, the solution of catalysts and methanol was clearly mixed with oil was fed to the microwave reactor using a peristaltic pump at different flow rate (6, 10.5 and 14.5ml/min). After the reaction in microwave oven in various microwave power output (40, 50, 60, 70 and 80% of maximum output power), the product was taken into a separatory funnel (250ml). At least three runs were conducted for reproducibility of biodiesel. After separation of glycerol layer, the top layer was centrifuged at 2500rpm for 20min to attain pure methyl ester phase from glycerol phase. Finally, the pure biodiesel was obtained by washing to remove traces of catalyst and heating to remove excess methanol or dissolved water in the mixture. Biodiesel yield (The composition of alkyl ester products) was calculated by GC (gas chromatography) analysis [15].

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**Table 1**: Properties of Waste cooking oil (WCO).

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Results and Discussion

Effect of methanol/WCO molar ratio

The in situ transesterification reaction of WCO was utilized. The pretreatment step (acid esterification prior to the actual base transesterification process) for removal of FFA in WCO was not performed due to time consuming and great loss of oil in washing step. Therefore, the pretreatment step was omitted and part of the KOH was used for neutralization of FFA and the remainder acted as transesterification catalyst. According to literature, Microwave irradiation is a well-established method of accelerating chemical reactions due to directly delivery the energy to the reactant. Effective heat transfer using microwave irradiation can be helped to complete the reaction in a much shorter time and obtaining higher yields in the production of biodiesel. Moreover, microwave irradiation reduced quantity of by-products. Obtained heat from microwave is result of dipolar rotation of polar solvents and the conductive migration of dissolved ions. Therefore, the methanol to WCO molar ratio is one of the crucial factors affecting reactivity. According to the chemical equilibrium, increasing the amounts of methanol can be accelerated the transesterification reaction to produce further biodiesel. On the other hand, excessive methanol amounts can reduce the concentration of catalyst and reactant, which slows the reaction and yield [9,33].

Figure 1: Effect of methanol/WCO molar ratios on biodiesel yield using 1wt.% KOH as catalyst and flow rate of 6ml/min.

Changes of temperature vs. time with increase the methanol/WCO molar ratio at different microwave output power are illustrated in Figure 2. It is observed that the reaction temperature increases faster by increase of the methanol/WCO molar ratio from 3 to 6 due to absorption of microwave irradiation with methanol as dipolar component. Moreover, it must be mentioned that the retention time reduced around 100 seconds. It is owing to increase local and bulk temperature of mixture due to dipolar rotation of ions and mixture receives as soon as to boiling point. Therefore, the mixture rate was increased in tube and exited faster [6]. However, the effect of reaction temperature was bigger than retention time. Therefore, the higher yield was obtained at the methanol/WCO molar ratio of 6.

On the other hand, it is known, when the molar quantity of methanol increased, the thermal gradient during the reaction was lower. Consequently, more energy is used for vaporization of alcohol and thus the rate of temperature increase is lower [29]. In addition, excessive methanol reduces the concentration of catalyst and reactant, which slows the reaction [33]. Moreover, this result could be related to insolubility alcohol of in triglycerides. There is a liquid-liquid process at the beginning of the transesterification reaction which control reaction rate by the diffusion of the reagents between the phases. If higher ratios use, the contact between the phases is probably not appropriate, and then, diffusion problems are greater and the yield decreases [29]. Therefore, the yields decreased for methanol/WCO molar ratio of 9 for all microwave output power. Consequently, methanol/WCO of 6:1 was chosen that the maximum yield was obtained at microwave output power of 400W (92.8%).

Effect of catalyst amount

It is explained above that polar components can absorb the microwave irradiation. The K+ and OH- ions existing in the feedstock mixture can be easily rotated with the high frequency electric field. Therefore, the OH- concentration results in faster reaction rate which increases with increasing amounts of KOH.
as catalyst. However, excessive amounts of catalysts will lead to soap formation, which increases the viscosity of the reactants and aggravates the separation of the products. Consequently, the amount of catalyst represents a critical parameter to obtain a high conversion yield [35]. In this study, transesterification was carried out using KOH as a catalyst at concentrations between 0.5%-1.5% of oil with the methanol/WCO molar ratio of 6:1, flow rate of 6ml/min and the various microwave irradiation output power. The results are depicted in Figure 3. When the catalyst concentration was set at 0.5% the yield was too low. The yield was specifically increased by increasing catalyst concentration to 0.75wt.%. However, a catalyst concentration of over 0.75% leads to saponification resulting in lower biodiesel yield and lower biodiesel quality [34]. At higher catalyst concentrations, the intensification of mass transfer is more important than increasing the amount of catalyst. Moreover, soap formation in the presence of high catalyst amounts increase the viscosity of the reactants and lowered the yield [35].

Figure 3: Effect of catalyst/WCO weight ratio on biodiesel yield using methanol/WCO mole ratio of 6 and flow rate of 6ml/min.

Figure 4 illustrates the change of reaction temperature with time for different microwave output power and catalyst consumption. As it has been discussed, it must be mentioned that the microwave irradiation would be absorbed by formed soap in high amount of catalyst loading. Encinar et al. [29] reported that dielectric constants of product mixture (methyl ester and glycerol) are greater than the content in reactant (triglycerides and methanol). Thus, the temperature significantly rises by the microwave radiation and the yield increase. The formation of soap decreases reaction rate and produced biodiesel. Therefore, the temperature does not adequacy raises. According to these results, we have chosen 0.75 wt.% KOH as a suitable catalyst concentration for the preparation of FAMEs from WCO that the yield of 96.5% was obtained at microwave output power of 450W.

Effect of flow rate

The transesterification was formulated as a three-step reaction. The triglyceride converts to diglyceride in first step by reaction with one molecule of alcohol. Then, the diglyceride converts to monoglyceride and finally, ester and glycerin prepare from reaction between monoglycerides and third molecule of alcohol. Hence, the transesterification time is important for completion of the reaction. Therefore, evaluation of an adequate transesterification time is necessary to guarantee completion of the reaction. In this work, it was assumed that there action occurs during microwave irradiation and reaction time was equal to its [36].

Figure 4: Temperature vs. time in conditions of 6 ml/min, methanol/WCO molar ratio of 6 and catalyst concentration of (a) 0.75 wt.% and (b) 1 wt.%.

Figure 5: Effect of flow rate on biodiesel yield using 0.75wt.% KOH as catalyst and methanol/WCO molar ratio of 6.

The experiments were carried out at different flow rate with 0.75wt.% KOH as catalyst and a methanol/oil molar ratio of 6:1 at various microwave output power. The results are shown in
Figure 5. When the flow rate increases then the yield of biodiesel slightly decreases. It could be reason of faster pumping of mixture from microwave oven and lower reaction time. Figure 6 shows the temperature vs. time with flow rate of 6 and 10.5ml/min. Due to faster pumping of reactants, the final temperature of mixture at flow rate of 10.5ml/min was lower than that at 6ml/min. The maximum temperature at flow rate of 10.5ml/min received to 42.2, 41.8, 43.3, 43.7 and 44.1 °C at microwave output power of 360, 450, 540, 630 and 720W, respectively. The reaction time also was lower 200 seconds. The temperatures are very lower than boiling point of alcohol. Therefore, the low temperature and time lead to less conversion of triglycerides to biodiesel [19]. However, the reaction time and temperature for flow rate of 6ml/min was higher at all microwave output powers. Consequently, flow rate of 6ml/min (retention time of 250 seconds) was selected as best flow rate for preparation of biodiesel. The results are also explained that microwave output power of 450W (50% of maximum microwave irradiation power) was the best irradiation power for biodiesel production in all flow rates according to EN standard which have good agreement with other researches [19].

**Figure 6:** Temperature vs. time in conditions of 0.75 wt.% catalyst, methanol/WCO molar ratio of 6 and flow rate of (a) 6 and (b) 10ml/min.

**Fuel properties**

The attained biodiesel in continuous system with microwave radiation in conditions of 450W microwave output power, a methanol/WCO molar ratio of 6:1, 0.75 wt.% of catalyst and flow rate of 6ml/min was analyzed. The properties of biodiesel such as density, viscosity, flash point, cloud point, pure point, acid value, and iodine value were determined as listed in Table 2. The values of prepared biodiesel show many similarities with the standard of biodiesel in Europe.

**Table 2:** Comparison of the properties of obtained biodiesel with the standard of Europe biodiesel.

| Parameters          | Unit          | Standard of Biodiesel (EN14214) | Obtained Biodiesel |
|---------------------|---------------|---------------------------------|-------------------|
| Density             | Kg.m$^{-3}$   | 860-900                         | 860               |
| Kinematic viscosity | mm2.s$^{-1}$  | 3.5-5.0                         | 3.88              |
| Flash point         | °C            | > 120                           | 172               |
| Cloud point         | °C            | -                               | 10                |
| Pure point          | °C            | -                               | 6                 |
| Acid value          | (mg KOH.g sample$^{-1}$) | < 0.5                         | 0.36              |
| Iodine value        | (g I2. 100 g sample$^{-1}$) | < 120                       | 79.9              |

**Comparison with literature**

The results of some studies on the biodiesel production via microwave irradiation were listed in Table 3. The biodiesel yield over 90% was achieved in all studies at similar methanol/oil molar ratios and catalyst concentrations. Although, they reported the maximum yield can be obtained at methanol boiling point temperature, the appropriate yield was attained at lower temperature (48 °C). The result was also better than discontinuous microwave-assisted biodiesel production from WCO as reported by Rahmanlar et al. [37]. The similar yield was obtained at lower duration while higher microwave output power and/or methanol was used [23,29-30]. However, higher microwave output power cause to more energy consumption and higher methanol consumption increases separation costs. From the comparison, it was clear that biodiesel yields obtained in this work were comparable to those reported in the literatures.
Table 3: Comparison of this study results with other literature reports.

| Feedstock | Temp. (°C) | Heating | Methanol/oil (mole ratio) | Catalyst (wt.%) | Time (min) | Microwave power (watt) | Yield | Ref. |
|-----------|-----------|---------|--------------------------|-----------------|------------|-----------------------|-------|------|
| WCO       | 48        | Continuous | 6                       | 0.75 (KOH)    | 4          | 450                   | 96.53 |      |
| Soybean   | 70        | Continuous | 12                      | 1 (KOH)       | 2          | 200                   | 96.5  | [29] |
| Jatropha  | 60        | Continuous | 6                       | 1 (CH₃ONa)    | 0.5        | 800                   | 96.5  | [30] |
| Palm      | 70        | Continuous | 12                      | 1 (NaOH)      | 1.75       | 400                   | 99.4  | [38] |
| Palm      | 70        | Continuous | 9                       | 1 (NaOH)      | 5          | 600                   | 99.8  | [39] |
| WCO       | 65        | Batch     | 6                       | 1 (NaOH)      | 5          | 600                   | 93.36 | [37] |
| Jatropha  | 60        | Batch     | 7.5                     | 1.5 (KOH)     | 2          | 1200                  | 97.4  | [23] |
| Cotton    | 60        | Batch     | 6                       | 1.5 (KOH)     | 7          | 250                   | 92.4  | [39] |

Conclusion

A microwave-assisted continuous flow system was adapted for the biodiesel production from WCO. The reaction conditions such as methanol/WCO molar ratio, catalyst amount and flow rate of reactant were assessed at various microwave output power. The results showed that the reaction time reduced to 3-7 min in the different conditions. The microwave irradiation obviously decreased the reaction time and presented high yield in all of conditions. The maximum yield (96.5%) was obtained at conditions of 6 molar ratio of methanol/WCO, 0.75 wt.% of catalyst and 6 ml/min of flow rate at 450W microwave output power. In these conditions, the reaction temperature was 48 °C. Although the mixture temperature did not receive to methanol boiling temperature, the local temperature probably was high enough for conversion of triglyceride and FFA to biodiesel. It could conclude that the heating by microwave irradiation was more effective for the biodiesel production by norm EN 14214 than conventional heating.

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