



Biodiesel production from waste cooking oil using calcined scallop shell as catalyst



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ABSTRACT

Transesterification of waste cooking oil (WCO) and methanol by using calcined scallop shell (CSS) as catalyst was carried out in a closed system for biodiesel fuel (BDF) production. It is found that the optimum calcination temperature for the preparation of CSS was 1000 °C. The effects of transesterification temperature, reaction time, methanol/oil molar ratio and catalyst loading amount on the BDF yield were investigated. Compared with the commercial CaO, CSS showed higher catalytic activity and the BDF yield reached 86% at 65 °C with a catalyst loading amount of 5 wt% (WCO basis) and a reaction time of 2 h. The catalyst was reused for 5 cycles whilst the BDF yield decreased 23%. It is found that CaO in CSS was transferred to calcium glyceroxide after the transesterification reaction, and calcium glyceroxide also showed good catalytic activity and reusability. Furthermore, Water content in WCO had negative effect on BDF yield. It is found that BDF yield reduced 15% due to the occurring of saponification when the water content was increased from 0.64% to 2.48%. It is expected that CCS can be used as an alternative and cheap catalyst for the biodiesel production.

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1. Introduction

The energy crisis and the impact of greenhouse gases to the earth arouse researchers to develop alternative energy for the replacement of fossil fuels. Biodiesel fuel (BDF) is an alternative fuel with eco-friendly because it is produced from natural resources. BDF is generally produced by the transesterification of vegetable oil or animal fat with alcohol such as methanol or ethanol in the presence acid or alkali catalyst [1–8]. The use of edible oils as sources of BDF will compete with the food and resulting in some social problems [7]. A source of BDF should have low cost and can be obtained easily around us because the major fraction of the BDF production cost is from the raw materials. In an area like Japan without enough natural resources, use of waste cooking oils (WCO) instead of original oils for the BDF production is an effective way to reduce the cost.

Catalyst is generally used for the transesterification reaction operated at ambient atmosphere. Various catalysts such as acid catalysts, base catalysts and enzyme catalysts are developed. However, acid catalyst presents low catalytic activity so that longer reaction time and higher reaction temperature are required [4–6]. Homogeneous alkali catalysts such as NaOH and KOH have good catalytic activity but they are difficult to be separated and reused after the reaction [7]. To solve this problem, heterogeneous alkali catalysts is used because they are non-corrosive, recyclable and effective. To date, various heterogeneous alkali catalysts have been developed for BDF production [8]. Among them, CaO is one of widely-used catalysts due to its abundant availability in nature, low cost and high activity [9–17]. CaO catalyst can be prepared from different source materials. For instances, it can be easily derived from lime stones and its derivatives, and waste shells such as waste egg shell [18,19], oyster shell [20], and scallop shell [21–23], which can not only save sources and decrease cost, but also solve environmental problems. However, these natural resources always come from some special regions. If they can be used properly for a regional sustainable development, it will be the most cost-effective way and good for the local society. In

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the North Japan area, a large amount of scallop shells (*Patinopecten yessoensis*) are produced every year, and the waste scallop shells are usually stacked on the road side, resulting in the air around it stink to high heaven. In order to use the waste scallop shell effectively and solve the regional environmental problem, in our previous studies [21–23], it was applied as a catalyst for the tar reforming in the biomass gasification process. It is found that calcined scallop shell (CSS) presents alkali property and contains special porous structure, and shows excellent catalytic activity for the tar reforming. To expand the utilization field of CSS as catalyst, in this study, CSS was used as a catalyst for the production of BDF from waste cooking oil (WCO). To increase the BDF yield, the calcination conditions and the transesterification conditions were optimized. Furthermore, the reusability of CSS and the effect of water content in WCO on the activity of CSS were also investigated. It is expected that CSS can be used as an alternative and cheap catalyst for the biodiesel production.

2. Experimental

2.1. Materials

In this study, WCO was provided by a local restaurant in Aomori City, Japan. This WCO contains 0.62 wt% of water with an acid value of 1.23 mgKOH/g and a saponification value of 212.2 mgKOH/g. Its density at 25 °C is 0.92 g/cm³. Methanol (99.5%), acetone, n-hexane and 2-propanol were purchased from WAKO, Japan. Waste scallop shell (WSS) was provided by Aomori City, Japan. Its main compositions are 98 wt% CaCO₃, 0.79 wt% MgCO₃ and 0.15 wt% SrCO₃. Various CSSs were prepared by crushing of WSS to powder and calcination of it at different temperatures of 600, 800, 1000, 1100, 1200, and 1400 °C for 2 h in air in order to understand the effect of calcination temperature on the catalytic activity. Since calcium glyceroxide is always generated during the transesterification reaction, herein, calcium glyceroxide was synthesized by heating the mixture of CSS, glycerol (WAKO, Japan) and methanol at 60 °C under mechanical agitation for 3 h [24]. For comparison, a commercial CaO (WAKO, Japan), which was calcined at 1000 °C for 2 h in air before use, was also used at the same reaction condition.

2.2. Transesterification reaction

As methanol is much more easily evaporated than triglycerides in the WCO, to make sure more methanol remained in the liquid phase to contact with triglycerides during the reaction, in this study, a closed reactor (50 cm³) was used instead of the reflux reactor. Prior to transesterification reaction, CSS and methanol were mixed in a closed reactor at 40 °C with a stirring speed of 500 rpm for 60 min. Thereafter, WCO was introduced into the reactor and transesterification reaction was carried out. Different methanol/oil molar ratios (3:1; 6:1; 12:1), catalyst loading amounts (1, 2, 5 and 10 wt% based on the oil weight), and reaction times (30, 60, 120 and 180 min.) were selected for the transesterification reaction. After the reaction, the product was centrifuged at 6000 rpm for 15 min. The upper ester layer was washed with deionized water and centrifuged again. This procedure was repeated for several times until pH value in the aqueous phase reached 7.0. Then, 0.1 mL of the product was mixed with 3 mL of n-hexane for the analysis. The concentrations of glycerides remained in the product and the produced fatty acid methyl ester (FAME) were analyzed using a high performance liquid chromatography (HPLC, Shimadzu, Japan) equipped with a silica-gel column (Shimpack CLC-SIL, Shimadzu, Japan) and a refractive index detector. The mobile phase was n-hexane/2-propanol at a ratio of 99.5/0.5 (v/v)

[3,5]. The column temperature was remained at 40 °C. Two peaks attributed to the unreacted glycerides (sum of mono-, di- and tri-glycerides) and the sum of FAMES appeared in the liquid chromatogram [25]. Thus, the FAME yield in the product can be calculated using the following equation:

$$\text{FAME yield} = (C_{\text{FAME}}/3C_{\text{oil}}) \times 100\% \quad (1)$$

where C_{FAME} and C_{oil} are the concentrations of FAME in the product and triglycerides in the WCO, respectively.

To investigate the effect of water content in WCO on the FAME yield, calculated amount of water was added in the WCO and mixed them homogeneously at first, and then they were used as WCO sources with different water contents (0.62, 1.24, and 2.48 wt% based on dewatered WCO). For the reusability test, the used catalyst was washed by acetone for 2 times and dried in vacuum, and then used again.

2.3. Catalyst characterizations

Morphologies of various CSSs obtained at different temperatures were characterized using a scanning electron microscope (SEM, SU6600, Hitachi). X-ray powder diffraction (XRD) analysis for the catalysts before and after the reaction was conducted using RIGAKU Smartlab X-ray diffractometer with Cu K_α radiation ($\lambda = 0.154$ nm) generated at 45 kV and 200 mA. Catalyst surface areas were measured using BET sorption isotherm method (Quantachrome NOVA 4200e, USA)

3. Results and discussion

3.1. Optimization of reaction conditions

Fig. 1 shows XRD patterns of CSS obtained at different calcination temperatures. One can see that all calcite in the original scallop shell converted to CaO completely at a calcination temperature of over 1000 °C. Thus, in this study, CSS obtained at 1000 °C calcination temperature was used at first for the optimization of reaction conditions. During the experiments, it is found that a reaction temperature of 65 °C, which is near the boiling point of methanol (64.7 °C at 1 atm), was the optimum temperature to obtain the highest FAME yield in all cases. In this study, a closed reactor was used, the pressure in the reactor should be higher than 1 atm, thus, most of methanol could remain in the liquid phase and increase the contact between oil and methanol. Therefore, all of the following data were obtained at the reaction temperature of 65 °C.

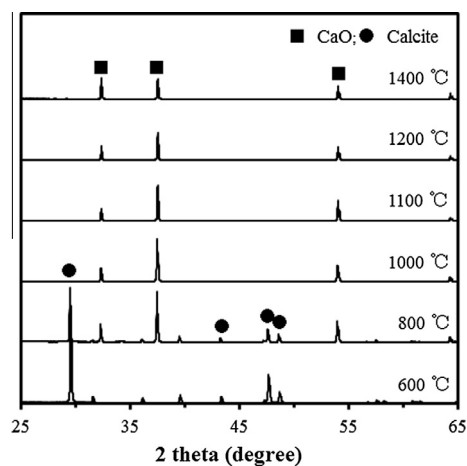


Fig. 1. XRD patterns of CSS obtained at different temperature.

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