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Improvement of the performance of encapsulated CaO and active carbon powders for rapeseed oil methanolysis to fatty acid methyl esters under condensed light irradiation

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ABSTRACT

The performance of capsulated CaO and active carbon powders for rapeseed oil methanolysis to obtain fatty acid methyl esters (FAME) under light irradiation was improved by inserting a condenser lens between the reactor and light source. Capsule breakage was greatly increased by the condenser lens because the lens increased the temperature inside the capsule dramatically. Consequently, a low FAME yield of 46.9% was obtained with the CaO (100 mg/mL)-active carbon (2.0 mg/mL)-loaded capsules after 6 h. Therefore, the effect of the irradiation power and active carbon loading on the final FAME yield was investigated. Decreasing the active carbon loadings from 2.0 to 0.1 mg/mL reduced the capsule breakage, which affected the final FAME yields. The decrease in the irradiation power from 3.6 to 0.6 W also affected the capsule breakage. An active carbon loading of 0.5 mg/mL and an irradiation power of 3.6 W gave the highest FAME yield (90%) after 6 h at 308 K. Under these conditions, the FAME phase only contained 0.37% Ca from the charged CaO catalyst with a capsule breakage of 53%.

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1. Introduction

The main component of biodiesel fuel (BDF), which is a possible carbon-neutral alternative to diesel, is fatty acid methyl esters (FAME). Therefore, much research has focused on triglyceride transesterification and free fatty acid esterification to synthesize FAME with various catalysts [1–9]. Currently, homogeneous acidic and basic catalysts are used for commercial BDF production from waste animal fat, non-edible oil, and edible oil; however, heterogeneous catalysts are preferred because catalyst recovery is easier and the BDF production procedure is simpler. We have investigated heterogeneous catalysts, particularly CaO, although during the reaction, the active species dissolve in the glycerol and FAME phases [10–21]. To address this problem, supported CaO catalysts [14,16–17] and binary mixture oxides [20] have been prepared that show improved stability, and the leached Ca has been removed in a purification step by an ion-exchange resin [22].

We have found that encapsulating CaO prevented the active species from dissolving, and that the activity of CaO-loaded alginate capsules remained the same after they were reused three times [23]. However, after the first run, capsule breakage of 20–40% was observed. Alginate capsules have been modified with silane coupling agents to solve this problem [24]. The rigidity of the CaO-loaded alginate capsule was

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Previously [25], rapeseed oil methanolysis was performed with CaOactive carbon-loaded alginate capsules under light irradiation, and it

photothermal material in the present study.

improved by successively forming alginate layers and modifying them with n-butyltrimethoxysilane. Yet, this modification slowed the perme-

ation of reactants, increasing the reaction induction and completion

times. These studies show that if the alginate capsule is used further,

the alginate shells will inevitably suffer from mechanical capsule break-

age by the stirring shear force. We also reported that the alginate cap-

sules containing CaO and active carbon powders exhibited a FAME

yield of about 80% after 6 h for rapeseed oil methanolysis with a xenon lamp as a light source [25]. Because this catalyst system was ap-

plied at low reaction temperature, the amount of Ca species that dis-

solved into the FAME phase was remarkably reduced. Moreover, the

visible and near-infrared (IR) light absorbed by the active carbon pow-

der heated the capsules. As the light-driven transesterification of rape-

seed oil was triggered by the photothermal exchange, the irradiation

power, which was determined by the light source, probably affected

the final FAME yield after 6 h [25]. To confirm this suggestion, we pre-

pared TiO₂-supported chromium oxide, which is a photothermal mate-

rial absorbs a broad range of irradiation like active carbon powder [26].

The results indicated that the power of the light irradiation absorbed by

Cr₂O₃/TiO₂ controlled the temperature inside the capsule, which deter-

mined the final FAME yield in the CaO-Cr₂O₃/TiO₂-capsule system. Be-

cause the photothermal properties of active carbon powder are superior to those of Cr_2O_3/TiO_2 , we chose active carbon powder as the







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was found that the low initial FAME yield (10%, 1 h) and insufficient final FAME yield (80%, 6 h) required improvement. Because the FAME yield is determined by the light irradiation power, in the present study the power was increased by inserting a condenser lens between the reactor and light source. Although the FAME yield could be improved by using the condenser lens, a rapid increase in the temperature inside the capsule probably caused higher capsule breakage during the reaction owing to the evaporation of low boiling point reagents such as methanol and tetrahydrofuran (THF). Thus, the effect of active carbon loading and irradiation power on the FAME yield was investigated to obtain the optimal conditions for the CaO-active carbon-loaded alginate capsules. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was used to estimate the amount of Ca species that had leached into the FAME phase after the reaction.

2. Material and methods

2.1. Materials

CaO powder (Wako Chemical Co., 7 m² g⁻¹) was used as received [24–26]. Sodium alginate, active carbon powder, and CaCl₂ were obtained from Kanto Chemical Co. THF (Kanto Chemical Co.), methanol (Kanto Chemical Co.), and rapeseed oil (Nacalai Tesque) were used in FAME synthesis. The rapeseed oil properties have been reported previously [23].

Methyl heptadecanoate (Tokyo Chemical Industry Co. Ltd.) and hexane (Kanto Chemical Co.) were used to prepare samples for analysis by gas chromatography-flame ionization detector (GC-FID). All other chemicals in this study were of analytical grade and were used as received.

2.2. Capsule preparation

The co-extrusion method was used to encapsulate the active carbon powder and CaO in an alginate shell with a coaxial needle with concentric nozzles [23–26]. An active carbon powder and CaO rapeseed oil suspension and aqueous 2% (w/v) sodium alginate solution were supplied by using syringe pumps through the inner and outer needle nozzles at flow rates of 7.5 and 50 mL h^{-1} , respectively. The two-phase droplets were caught in a 0.4 M CaCl₂ solution and stirred at 300 rpm for 30 min. After the capsules were recovered, they were washed with distilled water and dried in an oven for 10 h at 323 K before they were used in FAME synthesis. The CaO concentration was 100 mg/mL-oil, whereas active carbon concentrations were varied from 0.1 to 2.0 mg/mL-oil. The same procedure was used to obtain CaO-loaded capsules with different core suspensions. Fig. 1 shows microscope images of the capsules with different active carbon loadings. The shell thickness was about 30 µm for all capsules (Fig. 1(f)) and the diameter of the capsules was estimated to be about 2.0 mm from the microscope images (Figs. 1(a)-(e)). Although the shell thickness and diameter were similar, the core changed



Fig. 1. Microscope images of (a) a CaO-loaded capsule, (b) a CaO-AC (0.1 mg/mL)-loaded capsule, (c) a CaO-AC (0.5 mg/mL)-loaded capsule, (d) a CaO-AC (1.0 mg/mL)-loaded capsule, (e) a CaO-AC (2.0 mg/mL)-loaded capsule, and a cross-sectional SEM image of (f) a CaO-loaded capsule.

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