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NOTE

Preparation of Na_2SiO_3/CaO/KF Catalyst for Biodiesel Synthesis

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A nanosized Na_2SiO_3/CaO/KF solid base catalyst was prepared by grinding method. The characterized results showed that the new crystal $KCaF_3$ was formed in the catalysts. Under the optimal conditions, the biodiesel yield can reach 97 %.

Key Words: Solid base catalyst, Grinding method, Biodiesel.

Currently, transesterification reaction using base catalyst is the most extended process to produce biodiesel[1,2]. Low-cost catalysts that still retain the advantages of solid base catalyst should be exploited to simplify the preparation process. To our best of knowledge, there are no reports about the study of grinding method preparation of Na_2SiO_3/CaO/KF catalyst. In the present papers, solid base catalyst Na_2SiO_3/CaO/KF was synthesized by simply grinding method, the biodiesel yield can reach above 97 %.

Catalyst preparation: The Na_2SiO_3/CaO/KF (NCK) catalysts were synthesized by grinding method. A powder mixture of $Na_2SiO_3 \cdot 9H_2O$, CaO and $KF \cdot 2H_2O$ with the molar ratio of 1:2:x (where x represents the molar amount of KF, x = 0, 3, 6) was grinded, subsequently dried at 80 ºC for 8 h and calcinated at 400 ºC for 4 h. The as-prepared catalysts were denoted as NCK-x. The catalysts were characterized by using several techniques of the Hammett indicator, XRD, FT-IR, TG/DTA and TEM.

Transesterification reaction: The transesterification of soybean oil to biodiesel was performed in a glass bath reactor with a water-cooled condenser. Reaction conditions were as follows: molar ratio of methanol to oil was 10:1, the amount of catalyst was 3 wt %, reaction temperature was 65 ºC and reaction time was 0.5 h. The fatty acid composition of the product was then analyzed by gas chromatography using an internal standard[3].

Basic strengths of the catalysts: The basic strengths of NCK-x samples were examined. The base strength depending on amounts of KF, NCK-3 exhibited a maximum basic strength in the range of 15.0-18.4. The results suggest that strong base sites were hardly created with low KF amounts.

TG-DTA and XRD analysis: The TG-DTA curves of NCK-3 were shown in Fig. 1(a). No mass loss and an endothermic peak occurred in the range of 400-800 ºC, which suggested that crystal pattern transition lead to heat flow changes[4,5].

The XRD profile of the NCK-3 catalyst was shown in Fig. 1(b). The catalyst had the typical diffraction peaks of $KCaF_3$ crystal phase which was produced from a reaction between KF and the $Ca(OH)_2$ happened in the process of preparing catalyst[6].

FT-IR and TEM analysis: The IR spectra of NCK-x catalysts were shown in Fig. 2(a). For NCK-3 and NCK-6 catalysts, the absorption peak at 3647 cm^{-1} of $Ca(OH)_2$ disappeared with KF content increasing, but had a strong O-H band at 3350 cm^{-1}, which was assigned to the absorption of surface hydroxyl groups[7]. These results indicated the catalysts contain different types of basic sites[8].

The TEM images of the NCK-3 catalyst were shown in Fig. 2(b). The granules size of the bulk catalyst was distributed in the range of 30-100 nm. These results indicated that the as-prepared catalyst was a nanosized catalyst.

Transesterification over NCK-x catalysts: Under the same reaction conditions, biodiesel yield of the NCK-0, NCK-3 and NCK-6 catalysts was 46.4, 97.1 and 85.6 %, respectively. The maximum biodiesel yield was obtained over NCK-3. Therefore, NCK-3 was selected as the optimum catalyst for transesterification of soybean oil to biodiesel.

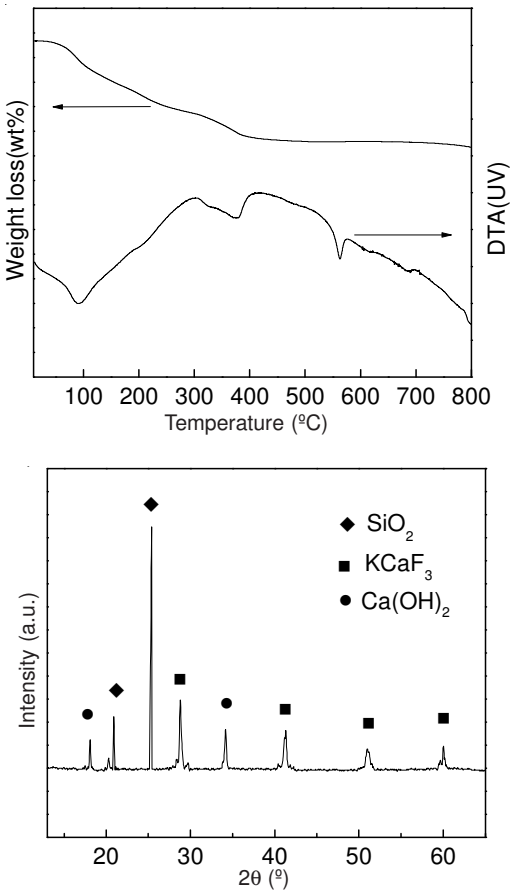
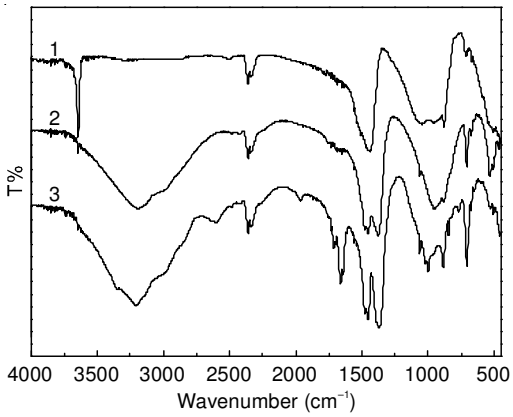


Fig. 1. (a) TG-DTA curves of NCK-3 catalyst precursor (b) XRD of NCK-3 catalyst



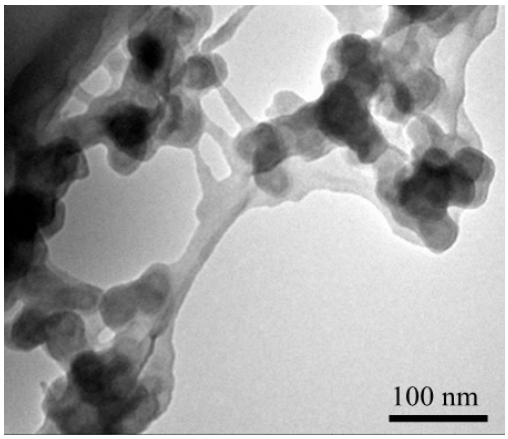


Fig. 2. (a) FTIR spectra of samples (1) NCK-0, (2) NCK-3, (3) NCK-6 and (b) TEM image of NCK-3 catalyst

Conclusion

A nanosized Na_2SiO_3/CaO/KF catalyst was prepared by grinding method. The highest fatty acid methyl ester yield can reach above 97 % over the NCK-3 catalyst. Through the XRD, FT-IR and TG-DTA analyses, the new crystal $KCaF_3$ was formed in catalyst, which enhanced catalytic ability.

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