Catalytic Cracking of VGO by Zeolite–kaolin Mixed Catalysts Using Curie Point Pyrolyzer

Atsushi ISHIHARA*, Kentarou KIMURA, Tadanori HASHIMOTO, and Hiroyuki NASU

Div. of Chemistry for Materials, Graduate School of Engineering, Mie University, 1577 Kurima Machiya-cho, Tsu 514-8507, JAPAN

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Mixed composite catalysts including three types of zeolite (Y, β and ZSM-5), kaolin and binder (Al2O3) were prepared as a model of an industrial catalyst for catalytic cracking and were tested in the catalytic cracking of VGO using a Curie point pyrolyzer (CPP) as a simple estimation method of catalysts for catalytic cracking. The mixed catalysts consisted of 26 wt% of zeolite, 58 wt% of kaolin and 16 wt% of a binder (Al2O3). The three mixed catalysts using Y (Si/Al2 = 5.5), β (37), ZSM-5 (90) zeolites were named MAT(kaolin)-Y, MAT(kaolin)-β and MAT(kaolin)-Z, respectively. MAT(kaolin)-Y and MAT(kaolin)-β showed higher selectivity for gasoline and higher RON value, and produced larger amounts of olefin and multi-branched products compared with single zeolite. These results indicated that both inhibition of over-cracking and dispersion of acid sites of zeolite were important to increase RON value. The SiO2/Al2O3 ratio of ZSM-5 used in this study was 90, and thus the density of acid sites of the mixed catalyst was much smaller than those for other zeolites used. Therefore, addition of kaolin to ZSM-5 significantly reduced the activity and the product selectivity. In this paper, the CPP method was found to be a simple and convenient tool to investigate the characteristics of catalysts for catalytic cracking of heavy feedstock such as VGO.

Keywords
Mixed catalyst, Zeolite, Kaolin, Catalytic cracking, Vacuum gas oil, Curie point pyrolyzer

1. Introduction

In recent years, crude oil production has included higher fractions of heavy oils such as vacuum gas oil (VGO) and atmospheric residue (AR). However, demand for gasoline and diesel oil has also increased due to further motorization in developing countries and emerging economies. Therefore, “catalytic cracking reaction” has become more important to increase production of gasoline and diesel oil by modification of the heavy oils. The characteristics of the catalysts used for catalytic cracking, such as fine pores of both the zeolite of the main catalyst and the matrix components, are thought to affect the catalytic activity and product selectivity. USY zeolite is generally applied for industrial processes, but other zeolite types have also been evaluated for catalytic cracking9–13). However, zeolites mainly contain micropores, which are not large enough to contain larger molecules such as VGO or AR or to allow the fast diffusion of substances, so matrices with mesopores have become the key catalyst components for fast diffusion of reactants and improvement of the catalytic activity and product selectivity in the cracking reaction. Nevertheless, few investigations of catalytic cracking have assessed the matrix components in contrast to the many studies of catalytic cracking using zeolite.

Recently, considerable attention has been focused on the preparation of meso-/microporous composite materials13–15). We have already reported that the combination of zeolite with micropores and matrix with mesopores promotes the formation of branched hydrocarbons, which mainly control the octane number of gasoline. The formation of novel hierarchical composite catalysts with both microporous zeolite and mesoporous matrices increased the selectivity for branched products with higher octane number because the large volumes within the mesoporous matrices around the active acid sites of the zeolite promoted the generation of more bulky branched hydrocarbons16–22). Furthermore, the catalytic cracking of VGO using a Curie point pyrolyzer (CPP) was shown to be a very simple and convenient method to determine the catalytic activity and selectivity for products19). On the other hand, the detailed catalytic reactivity and structural properties of industrial catalysts consisting of zeolite and matrices still remain unknown.

The present study investigated the relationship

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* To whom correspondence should be addressed.
* E-mail: ishihara@chem.mie-u.ac.jp

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between the catalytic activity and structural characteristics of mixed catalysts consisting of zeolite, kaolin, and alumina-sol binder as model catalysts of the industrial catalyst for catalytic cracking. The catalytic cracking of VGO using these model catalysts and the effects of three types of zeolite were evaluated using the CPP.

2. Experimental

2.1. Preparation and Characterization of Zeolite–kaolin Composite Catalysts

The types of zeolite used were ZSM-5 (MFI, JRC-Z-90H; SiO2/Al2O3 = 90, mean particle size (MPS) less than 5 μm, Catalysis Society of Japan), β-zeolite (BEA, HSZ-940HOA, SiO2/Al2O3 = 37, Al2O3 4.4 %, MPS 5 μm, Tosoh Corp.) and Y-zeolite (FAU, JRC-Z-HY5.5, SiO2/Al2O3 = 5.5, MPS 6 μm, Catalysis Society of Japan). The composite catalysts were prepared from zeolite (26 wt%), kaolin (Wako Pure Chem. Ind., Ltd.) as filler (58 wt%) and alumina sol (cataloid AP-1, JGC Catalysts and Chemicals Ltd.) as binder (16 wt%). All raw materials were mixed to prepare clay-like paste with continuous addition of small amounts of deionized water. Subsequently, the paste was press molded into pellets of 0.5 mm in diameter. Then, the pellets were heated to 600 °C at 2.4 °C/min and kept at 600 °C for 3 h. The pellets obtained were pulverized in a mortar before use. The composite catalysts were named as MAT(kaolin)-zeolite, where ZSM-5, β and Y zeolites were abbreviated to Z, β and Y, respectively.

The structural characteristics of the catalysts were measured by N2 adsorption (Bel Sorb mini, Bel Japan Inc.), using a calcined sample degassed under 10−2 kPa at 350 °C for 3 h, X-ray diffraction (XRD, Ultima IV), using nickel-filtered CuKα X-ray source radiation (λ = 1.5405 Å, 1 Å = 10−10 m) operated at 40 kV and 100 mA, and NH3 adsorption and desorption (GC-TCD, Shimadzu) with the pulse method; TPD of adsorbed NH3 was measured by heating the catalyst from 100 to 600 °C at a heating rate of 10 °C/min, according to the detailed methods described elsewhere14).

2.2. Catalytic Cracking of VGO Using a Curie Point Pyrolyzer

Catalytic cracking of desulfurized VGO (C 84.91 %; H 12.13 %; N 0.36 %, sulfur content: 100 ppm, supplied by Nippon Ketjen Co., Ltd.) was carried out using a CPP (JCI-22S, Japan Analytical Industry Co., Ltd.) under the conditions of 500 °C, catalyst 1.50 mg (particle size, 45-75 μm), and VGO 0.20 mg. Figure 1 shows the apparatus as well as the model structure of the catalyst. The CPP is a type of fixed bed reactor system, so can be substituted for the micro activity test (MAT) by choosing the appropriate reaction conditions. The samples of 0.20 mg of VGO and 1.50 mg of a catalyst were wrapped with pyrofoil specified for 500 °C (F 500, Japan Analytical Industry Co., Ltd.) and packed into the CPP. The pyrolyzer was preheated at 150 °C and an injection syringe was introduced into the injection port of GC. Pyrolysis at 500 °C for 5 s was performed under a stream of He at 0.45 MPa. All products were directly introduced into a GC-column and were determined by a hydrogen-flame ionization detector (FID) according to JIS K 2536-2 as specified elsewhere14). The conversion of VGO was estimated by comparing total area of products with that of standard gas references including C1-C4 (GL Sciences Inc.) according to the following equation.

\[
\text{Conversion of VGO} = 100 (\frac{PA \times C_{\text{GAS}}}{PA_{\text{VGO}} \times GA})
\]

where \(PA\) is the total peak area of products in VGO cracking measured by GC, \(C_{\text{GAS}}\) is the amount of carbon in the standard gas, \(C_{\text{VGO}}\) is the amount of carbon in VGO used, and \(GA\) is total peak area of the standard gas measured by GC.

3. Results and Discussion

3.1. Activity and Product Selectivity of Zeolite–kaolin Composite Catalysts in CPP Catalytic Cracking of VGO

Table 1 and Fig. 2 show the results from the catalytic cracking of VGO by zeolite–kaolin composites using the CPP. CPP proved to be a simple and useful tool for rapid analysis of the catalytic cracking of heavy oil compared to the widely used MAT method. Catalytic cracking of VGO using the CPP formed only small amounts of C1 and C2 products and thermal cracking hardly occurred, indicating that the VGO effectively diffused into the catalyst and that catalytic cracking of VGO proceeded selectively. The carbon numbers of the products for composite catalysts, MAT(kaolin)-β and MAT(kaolin)-Y, were higher than those of single β and Y zeolites (Fig. 2(a)). The NH3 adsorption-desorption data showed that the numbers of acid sites per unit weight decreased with the addition of matrix kaolin to retard over-cracking. No remarkable difference in the carbon number distribution could be seen for MAT(kaolin)-Z, indicating that single ZSM-5 with
The ratio of SiO₂/Al₂O₃ 90 provides adequate dilution of acid sites in a zeolite crystal, which effectively inhibits both over-cracking and change in the carbon number distribution of products. Figure 2(b) shows the selectivity for paraffins, olefins, naphthenes and aromatics by catalytic cracking products by zeolite-kaolin composite catalysts. Fewer paraffins and more olefins were formed by the composite catalysts compared with single zeolites, indicating that the hydrogen transfer reaction was inhibited by the reduction in acid sites per unit weight.

Table 1 shows the product distributions, characteristics of the gasoline fraction, and NH₃ adsorption-desorption data from the VGO cracking with zeolite-kaolin composite catalysts. The conversions of VGO were 29 % and 24 % using β and Y single zeolites, respectively, but the conversions slightly decreased using β and Y zeolite-kaolin composite catalysts. In contrast, ZSM-5 zeolite-kaolin composite showed much lower conversion than single ZSM-5 zeolite. The density of acid sites, as expected from the SiO₂/Al₂O₃ ratio, decreases in the order Y > β > ZSM-5. Therefore, coke formation and successive deactivation of active acid sites occurred due to the higher acid density of single Y and β zeolites, and the activity per acid site decreased for single zeolite and composite catalysts. The selectivity for gasoline increased and the selectivities for dry gas and C₁₂ products decreased using Y and β zeolite-kaolin composite catalysts. Presumably the decrease in the density of acid sites with the addition of kaolin inhibited over-cracking. The properties of the gasoline fraction showed higher ratios of olefin/paraffin (o/p) and multi/single (m/s) branched products for Y and β zeolite-kaolin composites. The o/p ratio increased since hydrogen transfer was inhibited by the lower acid density obtained by adding kaolin. β or Y zeolite is well known to have higher activity for hydrogen transfer. Consequently, the paraffin selectivity was much higher for single zeolite than for zeolite-containing catalysts. Furthermore, the selectivity for isoparaffins was the highest for single Y zeolite. The content of mesopores slightly increased with the use of composite as shown in Table 2. The increase in gasoline selectivity with lower acid density and the presence of mesopores promoted the increase in multi branched products for Y and β zeolite-kaolin composite catalysts. RON values for zeolite-kaolin composite catalysts were higher than those for single zeolites, possibly due to the higher olefin/paraffin ratios for the composite catalysts. Gasoline after catalytic cracking requires subsequent HDS to decrease sulfur content, so the improvement in RON value with high olefin/paraffin ratio may be lost. Since higher yields of branched products are required in

### Table 1

| Catalyst          | Products distribution [wt%] | Conv. [%] | Amount of NH₃ adsorbed [10⁻⁴ mol/g] | Parameters in gasoline fraction |
|-------------------|-----------------------------|-----------|-----------------------------------|---------------------------------|
| MAT(kaolin)-Z     | C₁-C₄: 52 G (C₅-C₁₁): 46 | 8         | 1.5 (0.3)⁵⁶                      | 1.94 2.12 0.16 104              |
| MAT(kaolin)-β     | C₁-C₄: 36 C₁₂: 60          | 4         | 3.6 (3.0)⁵⁶                      | 0.80 3.69 0.24 93               |
| MAT(kaolin)-Y     | C₁-C₄: 26 G (C₅-C₁₁): 71 | 4         | 4.4 (3.5)⁵⁶                      | 0.38 5.34 0.31 89               |
| ZSM-5 (90)        | C₁-C₄: 50 G (C₅-C₁₁): 48 | 2         | 10.0 (7.0)⁵⁶                     | 0.73 2.90 0.17 101              |
| Zeolite β         | C₁-C₄: 47 G (C₅-C₁₁): 51 | 2         | 8.9 (6.2)⁵⁶                      | 0.18 4.14 0.11 90               |
| Zeolite Y         | C₁-C₄: 36 G (C₅-C₁₁): 62 | 2         | 13.8 (11.7)⁵⁶                    | 0.12 7.33 0.12 87               |

a) Amount of NH₃ desorption.

### Table 2

| MAT(kaolin)-Z (90) | Products distribution [wt%] | Conv. [%] | Amount of NH₃ adsorbed [10⁻⁴ mol/g] | Parameters in gasoline fraction |
|-------------------|-----------------------------|-----------|-----------------------------------|---------------------------------|
| MAT(kaolin)-Z (90) | C₁-C₄: 52 G (C₅-C₁₁): 46 | 8         | 1.5 (0.3)⁵⁶                      | 1.94 2.12 0.16 104              |
| MAT(kaolin)-β     | C₁-C₄: 36 C₁₂: 60          | 4         | 3.6 (3.0)⁵⁶                      | 0.80 3.69 0.24 93               |
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a) Amount of NH₃ desorption.

**Fig. 2** (a) Carbon Numbers of Products from Catalytic Cracking and (b) Product Selectivity for Paraffins, Olefins, Naphthenes and Aromatics by Catalytic Cracking
modern FCC, the higher ratios of multi/single branched products obtained with zeolite-kaolin composite catalysts are important\textsuperscript{4(\textendash}22). Introduction of matrices into catalysts remarkably decreased the selectivity for paraffins but greatly increased the selectivity for olefins, indicating that the hydrogen transfer activity of the catalyst decreased simply because the content of zeolite had decreased. However, the activity of hydrogen transfer increased in the order of ZSM-5 $< \beta <$ Y in the zeolite-kaolin composite catalysts. The selectivities for aromatics were similar, indicating that the amounts of hydrogen formed during aromatic formation were not different in these composite catalysts. Therefore, this result may be related to the size of the micropores in the zeolite. Our previous investigation using hierarchical zeolite-containing catalysts had similar findings\textsuperscript{21}. Hydrogen transfer includes two molecular reactions. Y zeolite includes about 1.3 nm of supercages in its micropores which are not present in ZSM-5 and $\beta$ zeolite. Such supercages in Y zeolite could trap olefins with immediate formation of aromatics, which would lead to easy hydrogen transfer. Furthermore, the micropores are larger in $\beta$ zeolite than in ZSM-5.

### 3.2. Effects of Types of Zeolite in Catalysts on CPP Catalytic Cracking of VGO

Changes in the yields of gasoline, single- and multi-branched products with type of zeolite in the catalyst are plotted against the conversion of VGO with previous data using hierarchical zeolite-containing catalysts in Fig. 3\textsuperscript{31}. Higher conversion was correlated with higher yields, and clear linear relationships were observed in all these plots, although some deviations were also observed in the yield of multi-branched products. The plots for MAT(kaolin)-Y, MAT(kaolin)-$\beta$, and MAT(kaolin)-Z approximately lay on each line. These results indicated that the presence and properties of the matrix in the cracking catalysts greatly and fundamentally affected and increased all conversions, gasoline yield, and yields of branched products. Further, comparison of the data at the same conversion showed that the yield was approximately in the order HY series $>$ ZSM-5 series. The pore size and acid sites of the zeolite were correlated with these differences observed between types of zeolite and the structure of the catalysts including the matrix as well as the zeolite was the most important factor affecting the catalytic cracking activity and product selectivity. Both $\beta$ and Y zeolites have twelve membered rings at the entrance of the micropores whereas ZSM-5 has smaller ten membered rings. Furthermore, Y zeolite contains supercages, which would allow the formation of bulky carbenium ions and branched products. Therefore, the differences in yield at the same conversion are probably related to the size of micropores in the zeolite. The results for MAT(kaolin)-$\beta$, MAT(kaolin)-$\beta$, and MAT(kaolin)-Z confirmed these suggestions.

Kaolin-zeolite catalysts exhibited lower activity than hierarchical zeolite-containing mesoporous silica-aluminas as shown in Fig. 3, indicating that catalytic activity was greatly affected by the types of matrices even using the same zeolite, and that the introduction of larger numbers of mesopores into the catalysts increased the conversion and the yields of gasoline and branched products. However, kaolin is important in the catalytic cracking reaction as the presence of a matrix increases the dispersion of zeolite into the catalyst to decrease its acid density and to increase the activity and selectivity per unit zeolite in the catalyst.

### 3.3. Characterization of Zeolite–kaolin Composite Catalysts

ZSM-5, $\beta$ zeolite and Y zeolite were identified using the XRD data bases of JCPDS Nos. 0440003, 0560467, and 0882290, respectively. Figure 4(a) shows the XRD patterns of ZSM-5 zeolite, kaolin and the ZSM-5-kaolin composite catalyst (MAT(kaolin)-Z). Kaolin included crystals of quartz (27°, JCPDS 070344) and pyrophyllite (30°, JCPDS 0030170). The XRD peaks for ZSM-5 crystals were observed, indicating that the structure of ZSM-5 was maintained in MAT(kaolin)-Z\textsuperscript{31}. Similarly, XRD measurement confirmed that zeolite crystal structures were maintained in all MAT(kaolin)-zeolite samples. Furthermore, the weakening of the crystal pattern intensity seems to imply that the zeolite crystals are well dispersed in the matrix. As shown above, the mixed catalysts achieved relatively higher conversion although the catalyst contained only 26 wt% zeolite. This finding also confirmed the dispersion of zeolite crystals into the mixed catalyst.

Table 2 tabulates the N\textsubscript{2} adsorption-desorption results for zeolite-kaolin composite catalysts. The BJH results describe the findings of N\textsubscript{2} adsorption measurements obtained in the range from 3.3 to 200 nm pore diameter. Negligible adsorption was observed between 50-200 nm, so the BJH specific surface area and pore volume can be considered to reflect these values for the mesopores. The BET results represent the specific surface area and pore volume for all pores. BET surface
areas and pore volumes were lower for the composite catalysts than for the single zeolites. Such findings would result from the presence of pore-free kaolin and alumina binder with mesopores and reduced zeolite contents. Therefore, both micropores of zeolite and mesopores of alumina binder are present in zeolite-kaolin composite catalysts although the increases in BJH surface area were very small.

Table 1 and Fig. 5 show the amounts of NH₃ adsorbed and desorbed, and the NH₃-temperature-programmed desorption (TPD) profiles of zeolite-kaolin composite catalysts and single zeolites, respectively. The amounts of NH₃ adsorbed and desorbed for single Y zeolite were much smaller than the amount of Al contained in this zeolite, suggesting that the dispersion of Al was not so high in single zeolite compared to β zeolite and ZSM-518). The amounts of NH₃ adsorbed by composite catalysts were in the range 1.5-4.4 × 10⁻⁴ mol/g, which are approximately consistent with the decrease in zeolite content and shows that Al species present in the binder and kaolin did not form acid sites. Actually VGO cracking using only the binder or the kaolin did not form gasoline and light gas oil fractions which could be detected by GC. Therefore, only the zeolites contained the acid sites. All catalysts exhibited NH₃ adsorption and desorption. Although the relationships between the conversion of VGO and the amounts of NH₃ adsorbed and desorbed were not clear, both conversion of VGO and amounts of NH₃ decreased

(a) simple ZSM-5 (JCPDS No. 0440003), simple kaolin (quartz, JCPDS No. 0707344 and pyrophylite, JCPDS No. 0030170), ZSM-5-kaolin mixed catalyst; (b) simple β zeolite (JCPDS No. 0560467), β zeolite-kaolin mixed catalyst; (c) simple Y zeolite (JCPDS No. 0882290) and Y zeolite-kaolin mixed catalyst.

Fig. 4 XRD Patterns of Simple Zeolites and Zeolite-kaolin Mixed Catalysts
with lower zeolite content, indicating that the density of the zeolite acid sites in the mixed catalyst effectively decreased with addition of matrix kaolin to inhibit overcracking and coke formation. Specifically, the decrease in conversion was the most significant using the composite catalyst of ZSM-5, indicating that the dispersion of acid sites is higher in single ZSM-5 with SiO$_2$/Al$_2$O$_3$ ratio of 90 than in β and Y zeolites, and that most of Al species in single ZSM-5 form the active sites for the cracking reaction.

4. Concluding Remarks

The use of the CPP allows simple and rapid measurement of the characteristics of the catalysts. Kaolin–zeolite catalysts inhibited overcracking compared with single zeolite catalysts resulting in higher gasoline selectivity. Furthermore, higher olefin/paraffin ratio and multi-branched/single-branched hydrocarbon ratio in the gasoline fraction increased RON values. Although kaolin–zeolite catalysts exhibited lower activity than hierarchical zeolite-containing mesoporous silica–aluminas, kaolin acted as the matrix in the catalytic cracking reaction. The micro activity test (MAT) for catalytic cracking is generally used to evaluate catalysts$^{23)}$. However, the MAT is still somewhat large-scale for a university laboratory, so the utilization of CPP presented here would provide a simple alternative method for catalytic cracking of heavier resources. Although coke formation was not determined in this study, coke formation is the primary cause of catalyst deactivation in catalytic cracking$^{24),25)}$. Coke formation could be inhibited by forming nano-sized zeolites, with increased external surface area, indicating that even cracking of small molecules such as n-hexane would be affected by diffusion and the resultant coke formation and deactivation. Therefore, diffusion, coke formation and deactivation must have effects in our CPP method of VGO cracking. We are continuing the analyses of coke formation by thermogravimetry-differential thermal analysis (TG-DTA) although no reportable data has yet been obtained. These analyses will be reported to confirm the accuracy of this method.

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要 旨

キュリー・ポイント・パイロライザーを用いたゼオライト-カオリン混合触媒による VGO の接触分解

石原 篤，木村 健太郎，橋本 忠範，那須 弘行

三重大学大学院工学研究科分子素材工学専攻無機素材化学研究室，514-8507 滋賀県甲府市

本研究では、工業的に用いられる接触分解触媒のモデルとして、ゼオライトとカオリンからなる混合触媒を作製し、減圧軽油（VGO）の接触分解反応を試みた。反応装置では簡単な接触分解の評価方法として提案しているキュリー・ポイント・パイロライザー（CPP）を用いた。ゼオライト 26 wt%，カオリン 58 wt％，バインダー（Al2O3）16％を含有する混合触媒を、Y (Si/Al2 = 5.5)，β (37)，ZSM-5 (90) の 3 種類のゼオライトを用いて作製し、それぞれ MAT(kaolin)-Y，MAT(kaolin)-β および MAT(kaolin)-Z として表記した。MAT(kaolin)-Y と MAT(kaolin)-β では、ゼオライト単独を比較し、過分解を抑制して、ガソリン選択性を高めることができた。さらに、olefin/paraffin (o/p) 比，multi/single (m/s) 比，RON 値も増加させた。この結果は、RON 値を向上させる上で、ゼオライトを分散させ、酸点密度を低下させることで過分解を抑制することの重要性を示している。ZSM-5 は SiO2/Al2O3 が 90 であり、ゼオライト単独では他のゼオライトと比べて酸点密度が低く、カオリンとの混合により活性と選択性が大きく低下した。触媒評価は、通常、マイクロアクティビティテスト（MAT）が用いられるが、より短時間でより簡便に触媒テストを行う方法としてキュリー・ポイント・パイロライザー（CPP）を用いることができた。