

Synthesis of high surface area of hydrous zirconia and its catalytic activity for the dehydration of *iso*-propanol

SungWoo Baek¹, Eunpyo Hong¹,
Young Ho Lee², Chae-Ho Shin^{1,*}

¹Department of Chemical Engineering,
Chungbuk National University,
Chungbuk 28644, Korea

²Heesung Catalysts Corp., Siheung,
Gyeonggi 15088, Korea

*E-mail: chshin@chungbuk.ac.kr

Zirconium oxide has been applied to various catalytic reactions such as α -olefins from alcohol, 1-butene from 2-butanamine, hydrogenation of carbon dioxide, olefins and dienes. In many cases, ZrO₂ showed better catalytic performance than Al₂O₃ which is a widely used support [1, 2].

In this study, hydrous zirconia was synthesized in aqueous solution of zirconium oxychloride by a precipitation method with different precipitants: KOH, NaOH and NH₄OH. The solution was aged at 100 °C for various aging time, and then filtered and dried at 60 °C for overnight. The resulting hydrous zirconia was ion-exchanged in NH₄NO₃ solution to remove the residual Na⁺ and K⁺ ions. Finally, all samples were calcined at 700 °C for 6 h to obtain the crystalline ZrO₂. The samples denoted as P(x, y), where P, x, and y means the species of precipitant, aging time (h) and number of ion-exchange times, respectively.

The samples were characterized by X-ray diffraction (XRD), N₂-sorption analysis, scanning electron microscopy (SEM), *iso*-propanol-temperature programmed desorption (IPA-TPD), and Pyridine adsorbed infrared spectroscopy (Py-IR) to correlate with catalytic activity of the IPA dehydration.

Fig. 1 shows that pure tetragonal crystallites were formed from ZrO₂ catalysts aged for 24 h after calcination at 700 °C. From the N₂-sorption analysis, a KOH(24, 0) catalyst has larger specific surface area than NaOH(24, 0), and NH₄OH(24, 0). In addition, specific surface area of the samples was increased with the increasing aging time.

IPA-TPD results in Fig. 2 revealed that the NH₄OH(24, 0) had the lowest desorption temperature amongst the samples without ion-exchange process. However, ZrO₂ catalysts used KOH and NaOH after ion-exchange showed lower desorption temperature than NH₄OH(24, 0) catalyst.

The best catalytic activity for the dehydration of IPA was shown over a NaOH(120, 1) catalyst, and this result was well correlated with high specific surface area, and acid functionalities.

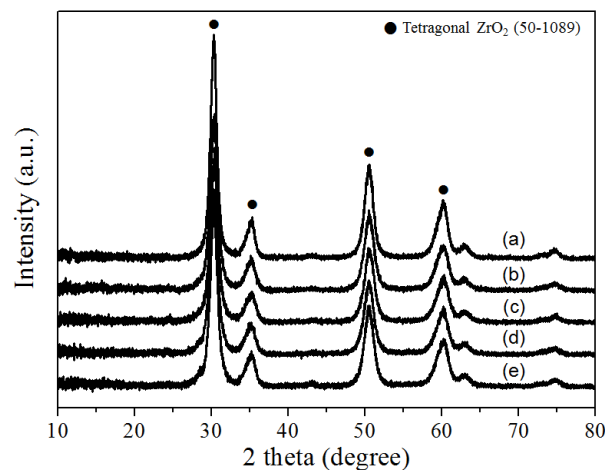


Fig. 1. XRD patterns of the calcined ZrO₂ catalysts aged for 24 h at 100 °C with different precipitants: (a) NH₄OH(24, 0), (b) NaOH(24, 0), (c) KOH(24, 0), (d) NaOH(24, 1), and (e) KOH(24, 1).

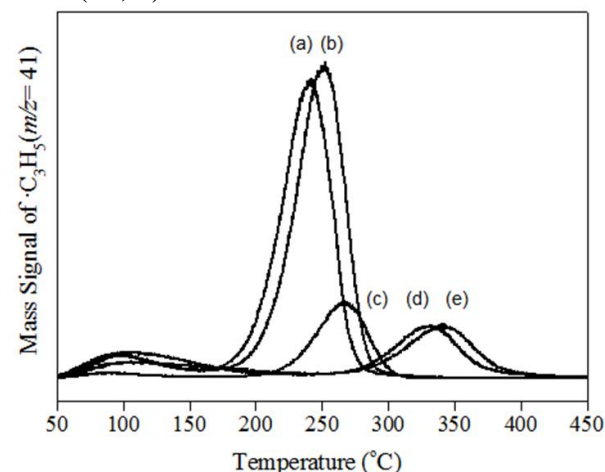


Fig. 2. IPA-TPD profiles of the calcined ZrO₂ catalysts. The mass signal of $\cdot\text{C}_3\text{H}_5$ ($m/z = 41$) was recorded by using QMS detector. Same notations in Fig. 1 were used.

REFERENCES

- [1] P.D.L Mercera et al. Appl. Catal. 71 (1991) 363.
- [2] K. Tanabe, T. Yamaguchi, Catal. Today 20 (1994) 185.