Application note (AN-2019-02EN)

Binary-component breakthrough curve measurement (CO_2/H_2O) using BELCATII

Introduction

In a variety of industrial processes, water vapor exists in low materials or as a by-product. The adsorption property of an adsorbent for objective gas component changes depending on the presence or absence of the water vapor, because of the competitive adsorption of water and objective component. Therefore, to design adsorption process, the adsorbent should be evaluated under the condition closer to the practical one, namely with the absence of water. In this article, a breakthrough curve measurement result of CO_2 and H_2O is described. The measurement was performed with our catalyst analyzer, BELCATII. The adsorption/desorption amount was quantified by using a quadrupole mass spectrometer, BELMass.

Measurement

As an adsorbent, zeolite molecular sieve 5A was filled in a triple structure sample tube of BELCATII, pretreated at 400 °C in helium flow, and a breakthrough curve measurement was carried out in CO_2/H_2O/He mixture (50 SCCM) flow. After this, the sample tube was purged with helium as a reactivation treatment, and TPD-measurement (temperature programmed desorption) was performed. The same measurement was performed for an empty sample cell. The adsorption/desorption amount in each process and the mass balance in the entire measurement were evaluated from the difference of each profile. To evaluate the concentration change of binary-component adsorptive, BELMass was used. The adsorption/desorption amount for CO_2 was calculated from the mass spectrum, m/z=44, and for H_2O from m/z=18.

Adsorbent:	Zeolite molecular sieve 5A (Amount: 0.1 g, particle size: 250 to 500 µm)
Pretreatment:	In 100% He flow (50 SCCM) At 400 °C, for 60 minutes.
Breakthrough curve measurement:	In a flow of CO_2 (1000 ppm)/H_2O (8000 ppm)/He (50 SCCM).
He purge:	In helium (50 SCCM), for 50 minutes at 25°C.
TPD:	In 100% He (50 SCCM) flow, at a 10°C min^{-1} ramp from 25 °C to 200 °C.

Result and Discussion

The continuous CO_2/H_2O breakthrough curve – TPD measurement result are shown in Figure 1. Comparing to H_2O, CO_2 reached the break point sooner. This indicates that the adsorption rate of CO_2 is higher than H_2O. On the other hand, from the fact that CO_2 concentration ratio became higher than unity within 1500 to 4000 seconds, it was considered that adsorbed CO_2 was desorbed by being replaced with in the binary-component breakthrough measurement. It was revealed that H_2O had a higher adsorption interaction than CO_2. This also can be confirmed by the fact that H_2O was not easily desorbed in the helium purge process, and desorbed completely after TPD measurement.

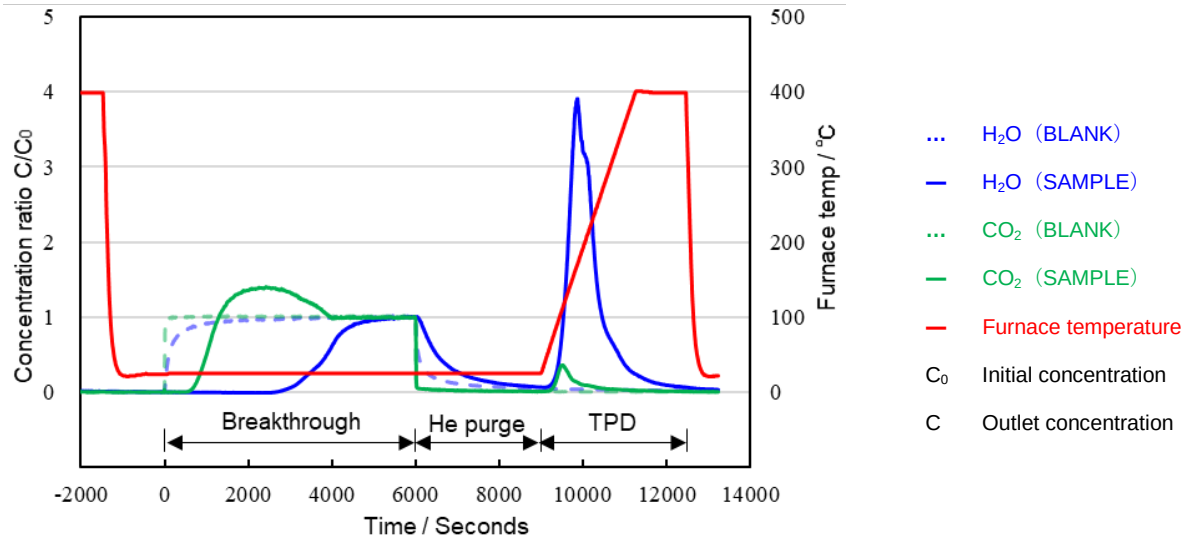

Fig.1 CO_2/H_2O Breakthrough curve-TPD measurement of zeolite MS-5A

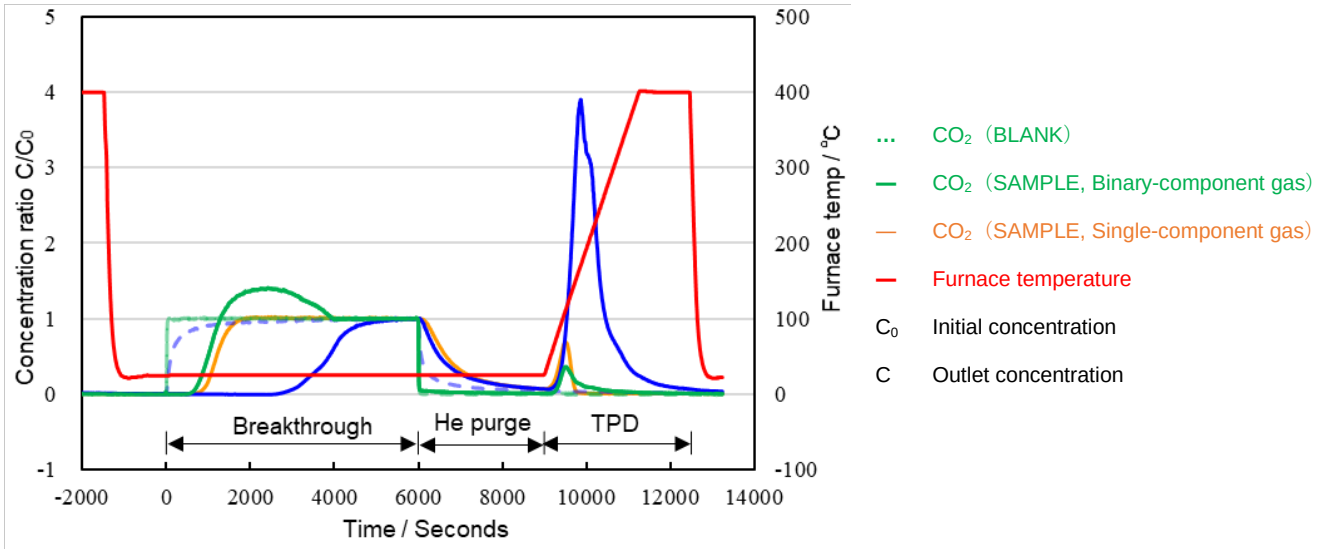

Fig.2 CO_2 Breakthrough curve-TPD measurement (Comparison of single/binary component measurement)

Table.1 Amount of desorption and Adsorption in each process (cm^3/g)

	Breakthrough (Ads.)	Breakthrough (Des.)	He purge (Des.)	TPD（Des.）	Mass balance*
Single-component -CO_2	9.45	—	7.62	1.97	101.5%
Binary-component -CO_2	7.48	5.53	0.07	1.52	95.2%
Binary-component -H_2O	219	—	29	174	92.7%

*Des.amount/Ads.amount ×100

The end point was reached in about 10 minutes. However, it took about 50 minutes for the reactivation process, and about 10% of CO_2 was desorbed during the TPD measurement. Therefore, it is supposed that there are some adsorption sites on which CO_2 can be adsorbed strongly, on MS-5A. In addition, as shown in Table 1, it can be seen that the adsorption amount of CO_2 in the two-component system was lower than the adsorption amount of CO_2 in the single component. The adsorption amount and the total desorption amount were balanced well. It was suggested that the reliability of the measurement was high.

Thus, CO_2/H_2O adsorption behavior was observed using BELCATII and BELMass. This function will be useful for the separation study.