

CO₂ breakthrough curve measurement using BELCATII

Introduction

The Adsorption breakthrough curve measurement is widely used as an assay method to examine design parameters and adsorption rates for adsorption process. In this article, aiming at single component gas recovery of CO₂, which is one of the greenhouse gases, we conducted CO₂ breakthrough curve measurement, and at the same time, helium purge and TPD measurement to observe the regeneration treatment process.



Experimental

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 1% - CO₂/He (50 SCCM) gas 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. The built-in thermal conductivity detector (TCD) was used as a detector.

Adsorbent:	Zeolite molecular sieve 5A (amount: 0.1 g, part. size: 250 - 500 μm)
Pretreatment:	In 100% He flow (50 SCCM) At 400 °C, for 60 minutes
Breakthrough curve measurement:	In 1% CO ₂ /He (50 SCCM) flow, at 25 °C, for 25 minutes
He purges:	In 100% He (50 SCCM) flow, at 25 °C, for 50 minutes
TPD measurement	In 100% He (50 SCCM) flow, at a 10°C min ⁻¹ ramp from 25°C to 200°C

Result and discussion

The continuous breakthrough curve – TPD measurement result are shown in Figure 1. The break point and the end point were observed about 5 minutes, and 10 minutes respectively from starting point of the breakthrough curve measurement. On the other hand, it took 50 minutes for the reactivation process, and about 10% of CO₂ was desorbed during the TPD measurement. It is considered that there might be some strong adsorption sites on MS-5A, on which CO₂ molecules can be adsorbed strongly.

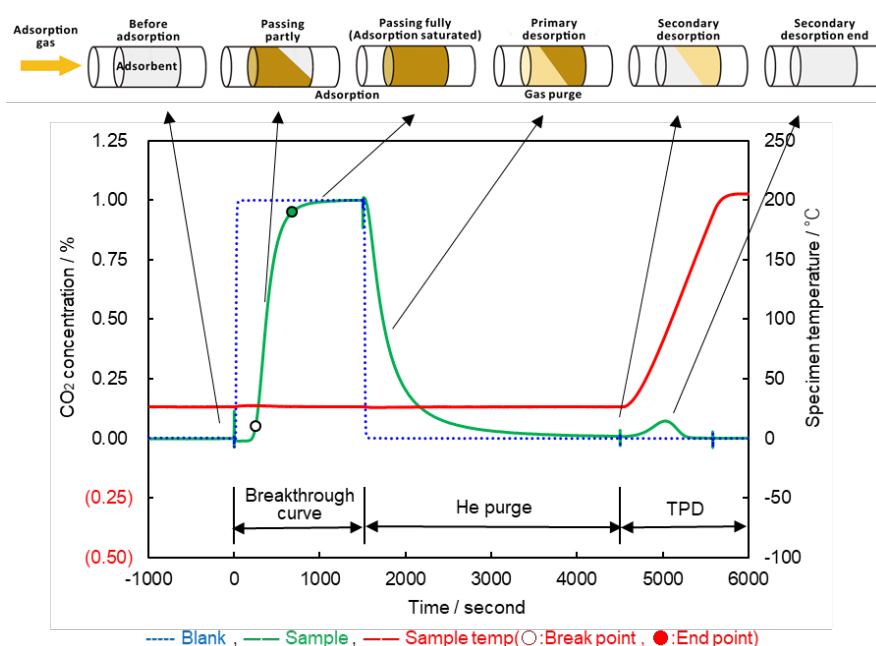


Fig. 1 CO₂ breakthrough curve-TPD measurement of zeolite MS-5A

Fig. 2 shows the difference between a blank sample measurement and a sample measurement result. From the shaded area of each peak, the adsorption/desorption amount can be calculated (Table 1).

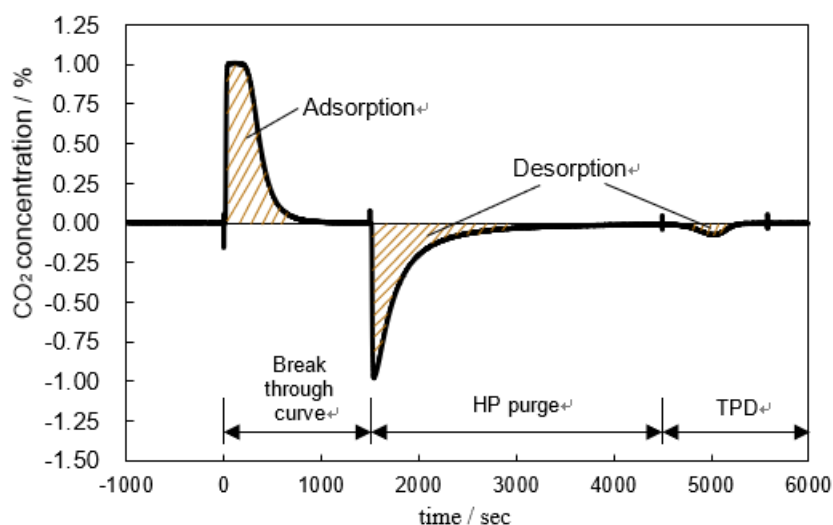


Fig. 2 Difference between blank and sample measurement

Table 1: Amount of adsorption and desorption in each process

Breakthrough curve(Ads.)	He purge(Des.)	TPD(Des.)
Ads.amount: 1.41 mmol/g	Des.amount: 1.26 mmol	Des.amount: 0.12 mmol/g
Break point (Saturation degree 5%): 250 seconds		Peak top temp.: 90°C
End point (Saturation degree 95%): 680 seconds		

Mass balance : $(1.26+0.12) / 1.41 \times 100 = 97.9\%$

Thus, by the continuous breakthrough – He purge – TPD measurement, the adsorption amount and desorption amount (reactivation process) of adsorbents can be evaluated quantitatively.

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