

§ 8. Experimental Analysis of Hydrogen Adsorption for Isotope Separation/Purification by Pressure Swing Adsorption

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In order to perform the LHD deuterium experiments, a practical system of hydrogen isotope separation is necessary for the environmental safety treatment of exhaust gases. We are scheming to apply the pressure swing adsorption (PSA) to the isotope separation, and have clarified the equilibrium characteristics of multi-component adsorption of hydrogen isotopes on a candidate adsorbent, synthetic zeolite 5A-type, in the previous year's study.

In the present study, a multi-component gas analyzing system is developed to measure breakthrough hydrogen isotopes with an adsorption column, and then fundamental experiments are carried out for examining the breakthrough characteristics of tracer D_2 in a H_2 - D_2 mixture with a zeolite 5A-type packed-bed column at a cryogenic temperature of 77.4 K. Experimental results are theoretically analyzed.

i) Development of inline gas-analyzing system

The hydrogen isotope gas analyzing system developed in this work is composed of a quadra-pole-type mass spectrum analyzer mounted on a high vacuum chamber with a turbo-pump, and a two-stage sample splitter. This device can split an infinitesimal molecular stream from an atmospheric gas stream and introduce it into the analyzer's chamber, through a pressure reducing stage of sample gas. Experiments for calibration demonstrate that this system can analyze ppm-order D_2 in a hydrogen isotope mixture with responsiveness.

ii) Breakthrough experiment with adsorption column

Figure 1 shows examples of experimental breakthrough curves. Breakthrough behavior of D_2 in a hydrogen gas flowing through a synthetic zeolite packed-bed column at 77.4 K is examined by using a cryogenic PSA apparatus in the NIFS. The test column is used of $\phi 40$ mm in inner diameter, where adsorbent particles of $\phi 2$ mm in mean diameter are charged at an amount of 700 g on a dry basis. The hydrogen mixture including D_2 at a concentration of 10,000 ppm is used in this experiment.

By evacuation using a scroll-type vacuum pump, the desorption process is operated at the same temperature as the adsorption. The evacuating operation is carried out for 20 minutes while the evacuation pressure reaches around 2,000 Pa and a volume adsorbed at the atmospheric pressure is reduced to 30 %. The column evacuated is filled with pure hydrogen by introducing from its outlet, before the adsorption process. In experimental observation, it is confirmed that breakthrough curves are reproductive during

several repeats of operation between adsorption-desorption processes.

iii) Breakthrough curve analysis

The amount of H_2 adsorbed at the atmospheric pressure, obtained from a preparatory breakthrough experiment of H_2 with an original column filled with helium, almost agrees with the value estimated from the isotherm for H_2 on zeolite 5A-type shown in the previous report.

The amount of D_2 adsorbed which is calculated from a breakthrough curve is consistent with the value estimated from an isotope separation factor of D_2/H_2 obtained in the previous study, based on the ideal adsorbed solution theory. The result shows that a separation factor around 2 for D_2/H_2 is expectable in this experimental adsorption system.

Breakthrough curves in adsorption systems exhibiting the Henry type of isotherm can be described with an equation derived from a theoretical model. Since this experimental system is of an isotope molecular exchange reaction which exhibits the Henry type adsorption, obtained breakthrough curves of tracer D_2 carried on bulk H_2 are crossly simulated by theoretical curves.

In this experimental study, valuable knowledge and data are obtained, and fundamental but important factors for considering the design and operation of a PSA process for hydrogen isotope separation are clarified from the curve fitting analysis.

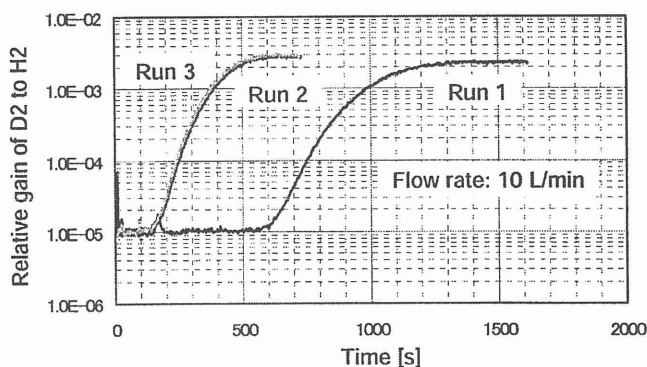


Fig. 1 Breakthrough curves of D_2 in a hydrogen mixture with a zeolite 5A-type packed-bed column

References

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