One-pot synthesis of HKUST-1 monolith for CO₂ adsorption CO₂吸着用 HKUST-1 モノリスのワンポット合成 (早大ナノ・ライフ^{*}・早大先進理工^{**}・ENEOS 株式会社^{***}・早大理工総研^{****}) ○酒丼款^{**}・堀崔太^{**}・伊藤綾春^{**}・松茶隆⁴^{***}・朝野 副^{***}・松芳琵^{*},**,****

1. Introduction

Metal-organic-framework (MOF) is drawn attention as CO₂ adsorbent. Shape forming technology for powdery MOF is required in order to generate objects of millimetric dimensions with enough mechanical resistance for application in industry¹). In this study, alumina tube and Cu cube were used as the metal source for MOF preparation and a part of them was directly converted into MOF monoliths. The preparation method of MOF monolith and its adsorption property were investigated.

2. Experimental

Two types of monoliths containing MOFs, HKUST-1 and MIL-96, were prepared. HNO₃, trimesic acid (TMA), and porous α -alumina tube were used as raw materials for MIL-96 preparation. Porous Cu cube was used for HKUST-1 preparation instead of α -alumina tube. Either alumina tube or Cu cube was placed into the aqueous solution of HNO₃ and TMA, and then hydrothermally treated by using glass-lined autoclave. The effect of these preparation conditions on the preparation of MOF monolith was studied.

MIL-96 powder was synthesized according to the literature²⁾ as reference material. HKUST-1 powder (Basolite C-300) was purchased from Sigma-aldrich.

CO₂ adsorption properties of prepared MOF powder and monoliths were measured by volumetric adsorption method (Belsorp-MAX, microtracBEL).

3. Results and discussion

The concentrations of HNO₃ and TMA and the conditions of hydrothermal treatment were widely changed. By the optimization of synthesis conditions, the contents of MIL-96 and HKUST-1 in monoliths increased up to 6.0 and 32 wt%, respectively.

Fig. 1 shows the morphological features of raw materials of metal sources and monoliths synthesized. The surface of alumina tube was fully covered with

MIL-96 crystals and the color of alumina tube changed to yellow. The surface of Cu cube was converted to HKUST-1 as well.

Fig. 2 shows the isotherms of CO_2 on MIL-96 and HKUST-1 monoliths and powders at 298 K. The adsorbed amount of CO_2 at 100 kPa on MIL-96 monoliths and powder were 140 and 95 cm³(STP) g⁻¹, respectively. HKUST-1 powder and monolith showed almost the same values of the CO_2 adsorbed amount, 99 cm³(STP) g⁻¹.

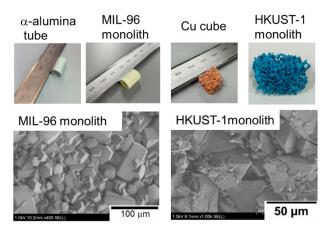


Fig. 1 Typical photo and FE-SEM images of α -alumina, MIL-96 monolith, Cu cube and HKUST-1 monolith.

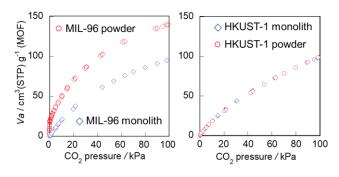


Fig. 2 CO₂ isotherms on MIL-96 and HKUST-1 at 298 K.

References

1) Bazer-Bachi, D., *et al., Powder Technol.*, **255** (2014) 52-29.

2) Benoit, V. et al., J. Mater. Chem. A, 6 (2018) 2081-2090.