

§1. Preparation and Characterization of Porous Polymer Supported Pt Catalyst

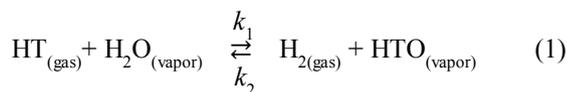
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For the detritiation from tritiated water by the water-hydrogen chemical exchange system, the use of isotope exchange catalyst is thus indispensable. However, as well known, such a catalyst (known as the Kogel catalyst) is difficult to produce and relatively expensive. Therefore, there still is a need to develop a novel inexpensive catalyst with good reproducibility. In this study, a novel porous styrene-divinylbenzene (SDVB) polymer was chosen as the catalyst support, and the control of the pore size in the range from about 2,000 to 100 nm was examined. The active component Pt was loaded into the obtained SDVB polymer, and the catalytic activities in H-T chemical exchange were investigated.

Macroporous SDVB polymers were prepared from the mixture of styrene (1.2 mL), divinylbenzene (1.8 mL), 1,3,5-trimethylbenzene (4.2 mL), 2,2,6,6-tetramethylpiperidine 1-oxyl, benzoyl peroxide (0.03 g), acetic anhydrous (0.02 mL), and dimethylpolysiloxane (denoted DMS), according to the literature with a slight modification.¹⁾ The SDVB polymers obtained using 0.36, 0.34, 0.30 and 0 mL of DMS were denoted as No. 1, No.2, No. 3 and No. 4, respectively. Pt loading (1.0 wt%) on the porous SDVB polymer was performed by impregnating $\text{H}_2[\text{PtCl}_6] \cdot 6\text{H}_2\text{O}$ in an ethanol solution. Pt was reduced at 200 °C in an H_2 (50%)/ N_2 (balance) flow before use in the catalytic reaction.

A continuous network of wormhole-like macropores were confirmed in No. 1, 2 and 3 by the FE-SEM measurements, however, such porous structure was not seen in No. 4 (Fig. 1) The macropore diameter was determined to be 2,361 (No. 1), 522 (No. 2) and 102 nm (No.3), respectively, by Hg porosimetry. In contrast, No. 4 did not have a distinctive macropore in the range above 50 nm. These results indicate a successful control of macropore diameter depending on the amount of DMS used.

Gas-phase T exchange between H_2 and tritiated water (eq.(1)) was performed using a conventional liquid-phase



chemical-exchange column reactor (inner diameter 25 mm). H_2 gas flow (2–7 NL/min) with a saturated tritiated water (about 4,000 Bq/mL) was passing the catalyst bed (about 2 g of Pt/SDVB catalyst) at 70 °C.²⁾ The effluent HTO was collected using a water bubbler (5 °C) or a cold trap at -100 °C, and its concentration was determined using a liquid scintillation counter (Aloka, LSC5100). The catalytic activity was evaluated by the overall mass transfer coefficient k (s^{-1}) from the following eq. (2).

$$\ln(1 - \eta) = -(k_1 + k_2) t_0 = -kt_0 \quad (2)$$

where η (-) is the relative reacted ratio. k_1 and k_2 are the apparent rate constants for the forward and reverse reactions, respectively.

In Fig. 2, plots of the changes in k as a function of the SDVB macropore diameter are shown. The plots clearly show that k depends on the macropore diameter, and the maximum k occurred at around 500 nm. These findings will be valuable for the development of novel catalysts for hydrogen isotope chemical exchange reactions.

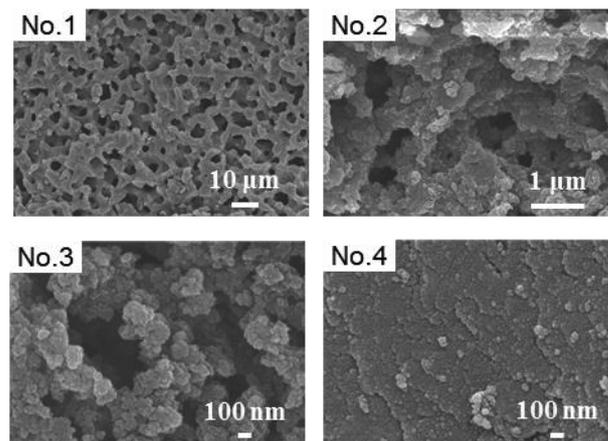


Fig. 1 FE-SEM images of SDVB polymers prepared.

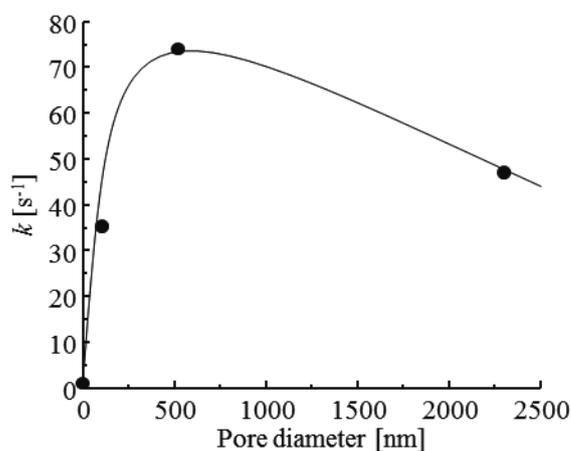


Fig.2 Changes in k as a function of SDVB pore diameter.

- 1) Kanamori, K. et al.: Adv. Mater. **18** (2006) 2407.
- 2) Izawa, H. et al.: J. Nucl. Sci. Technol. **16** (1979) 741.
Sugiyama, T. et al.: Fusion Sci. Technol. **60** (2011). 1323.