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Unique dissolution/precipitation phenomena in microspace induced by physisorption

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In this study, we used mesoporous silica MCM-41 as the vessel of the micro-solution, dibenzyl as solute, and water and diethyl ether as solvents. The water adsorption isotherm of dibenzyl pre-adsorbed MCM-41 (MCM-Dib) shows a new type of isotherm shape which not classified by IUPAC. The water adsorption amount is independent of the amount of dibenzyl (ϕ_Dib) and agreed with the pore capacity of a raw MCM-41. We confirmed by DSC, wide-angle X-ray scattering and NMR measurement that the precipitation of dibenzyl occurs outside of the pores at second step of the water isotherm. In this micro-solution system, opposite to the bulk, the solute precipitates as the amount of solvent increases. In the case of diethyl ether, the adsorption amount increased specifically at high relative pressure, reaching 3.5 times that of a raw porous solid. This increase in adsorption is attributed to the formation of the new solution phase on the outside of the pores. These results indicate that the combination of dibenzyl and water, which is insoluble in the bulk, is soluble in the pore. The micro-solution formed in the pores is promising as a new reaction field, and their unique reverse dissolution and precipitation behavior is expected to be used for drug delivery and other applications.

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