**In situ X-ray Diffraction Study of High-Pressure CO\(_2\) Adsorption in Metal-Organic Frameworks**

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The adsorption of CO\(_2\) onto metal-organic framework (MOF) compounds leads to a series of structural changes driven by the incorporation of gas molecules into the crystal voids. We report an *in situ* powder x-ray diffraction study of MOF compounds as a function of two sets of parameters. First, the dependence of the steady-state structure on CO\(_2\) pressure was used to search for a threshold pressure for the expansion of the lattice. We focus in particular on the expansion of the spacing of the (020) planes which are sensitive to the inter-sheet spacing in the MOF. A second time-dependent study is used to probe the adsorption kinetics. The experiments used a specially designed gas delivery system incorporated into a four-circle diffractometer with a rotating anode x-ray generator. The setup allowed precise control of the gas atmosphere during diffraction experiments, with gas pressures up to 50 atm. The experimental results provide insight into the structure of CPL-2 (Cu\(_2\)(pzdc)\(_2\)(bpy); pzdc = pyrazine-2,3-dicarboxylate) and CPL-5 (Cu\(_2\)(pzdc)trans-1,2-bis(4-pyridyl)ethylene) during CO\(_2\) adsorption-desorption cycles. A set of control experiments was conducted using a N\(_2\) environment in the same range of pressures. The experiments reveal an expansion in the inter-sheet spacing in CPL-2 and CPL-5 that increases over the full range of applied pressure, up to 50 atm, with CPL-5 exhibiting nearly 18% more expansion than CPL-2 at 50 atm. Results from the kinetics experiments indicate rapid CO\(_2\) uptake in both CPL-2 and CPL-5, with approximately 90% of the inter-sheet expansion occurring within the first three minutes and the remaining expansion occurring over a range of several hours.