308h Removal of the Structure-Directing Agent from a Thermally Unstable Cobalt Substituted Aluminophosphate

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Conventional molecular sieves comprise centers of silicon, aluminum, and phosphorous. However, it is possible to incorporate other metals into the framework creating sorbents of relevant industrial interest. The challenge is to synthesize new crystalline nano-porous materials with specific framework topologies and novel compositions based on templated hydrothermal paths. Extensive research for the development of ultra-large pore structures have produced materials like VPI-5 and ALPO4-8, which have pore windows of 18- and 14-member rings, respectively. However, both materials have frameworks with channels with the largest pore in one dimension and do not have extra-framework metal cations because their structure charge is neutral. There is a new type of high surface area molecular sieves based on transition metal substituted aluminophosphates known as UCSB-8Co (SBE). SBE is a large-pore material with unique framework characteristics and has the following composition $|(C_9H_{24}N_2^{2+})_{16}|$ $[Al_{32}Co_{32}P_{64}O_{256}]$, where $C_9H_{22}N_2$ (1,9-diaminononane) is the structure-directing agent (SDA) used during synthesis. These molecular sieves have channels with 12-ring apertures in 2-D and 8-ring apertures in 1-D, with supercages measuring 20Å by 20Å by 15Å. Such cage geometry is rather unusual when compared to that of faujasite cages, which are of spherical type but measure only 13Å in diameter. The characteristics of UCSB-8Co sorbents are ideal for adsorption applications where both specific sorption interactions and large void volume are critical. Nevertheless, the high transition-metal concentration contributes to the lower thermal stability of UCSB-8Co as compared to other Al^{3+}-rich molecular sieves. Conventional methods for removing organic templates from molecular sieves involve the use of air at high temperatures to break down and desorb the template via Hoffman elimination. Preliminary tests done by our group showed that a calcination process in oxygen-only atmosphere further damaged the SBE material framework. Another SDA removal test using solvent extraction methods demonstrated to be capable of recovering some void space in the micropore region, which indicates that not all template molecules are protonated, as previously thought. Other techniques for removing SDAs include low-temperature calcination by ozone and/or ion exchange. For this work, the latter will be used coupled to solvent extraction methods to achieve complete removal of the protonated and non-protonated moieties. The removal of the organic template after synthesis is essential to release the void space necessary for adsorption of guest molecules and allowing ion exchange whereas preserving the framework integrity.