Chemical Engineering Thesis Defense

Synthesis, Characterization and Oxygen Adsorption Properties of Substituted Aluminophosphate (AlPO4-5) Zeolites

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Abstract

The objective of this research was to develop Aluminophosphate-five (AlPO4-5, AFI) zeolite adsorbents for efficient oxygen removal from a process stream to support an ongoing Department of Energy (DOE) project on solar energy storage. A molecular simulation study predicted that substituted AlPO4-5 zeolite can adsorb oxygen through a weak chemical bond at ambient temperature. Substituted AlPO4-5 zeolite was successfully synthesized via hydrothermal crystallization by following carefully designed procedure to tailor the zeolite for efficient oxygen adsorption. Synthesized AlPO4-5 in this work included Sn/AlPO-5, Mo/AlPO-5, Pd/AlPO-5, Si/AlPO-5, Mn/AlPO-5, Ce/AlPO-5, CuCe/AlPO-5, and MnSnSi/AlPO-5. While not all zeolite samples synthesized were fully characterized, selected zeolite samples were characterized by powder x-ray diffraction (XRD) for crystal structure confirmation and phase identification, and nitrogen adsorption for their pore textural properties. The Brunauer-Emett-Teller (BET) specific surface area and pore size distribution were between 173 m2/g - 306 m2/g and 6Å - 9Å, respectively, for most of the zeolites synthesized. Samples of great interest to this project such as Sn/AlPO-5, Mo/AlPO-5 and MnSnSi/AlPO-5 were also characterized using x-ray photoelectron spectroscopy (XPS) for elemental analysis, scanning electron microscopy (SEM) for morphology and particle size estimation, and Electron Paramagnetic Resonance (EPR) for nature of adsorbed oxygen. Oxygen and nitrogen adsorption experiments were carried out in a 3-Flex adsorption apparatus (Micrometrics) at various temperatures (primarily at 250C) to determine the adsorption properties of these zeolite samples as potential adsorbents for oxygen/nitrogen separation. Experiments showed that some of the zeolite samples adsorb little-to-no oxygen and nitrogen at 250C, while other zeolites such as Sn/AlPO-5, Mo/AlPO-5, and MnSnSi/AlPO-5 adsorb decent but inconsistent amounts of oxygen with the highest observed values of about 0.47 mmol/ g, 0.56 mmol/g, and 0.84 mmol/ g respectively. The inconsistency in adsorption is currently attributed to non-uniform doping of the zeolites. However, more investigations are needed to verify the causes of this inconsistency to develop a successful AlPO4-5 zeolite-based adsorbent for oxygen/nitrogen separation. These findings validate that some substituted AlPO4-5 zeolites are promising adsorbents but a decision remains to be made to either improve those that exhibited decent oxygen adsorption amounts or evaluate alternatives that require no doping.

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