

HIERARCHICAL ZEOLITES FROM *TOP-DOWN* APPROACH: A SYSTEMATIC INVESTIGATION OF THEIR STRUCTURAL AND ADSORPTION PROPERTIES

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Hierarchical zeolites, which exhibit a distinctive structure characterized by interconnected micropores and mesopores, are versatile materials that can overcome the typical issues related to microporous materials, including poor mass transfer, hindered diffusion, and strict selectivity of resulting products. One possible route to obtain hierarchical porous materials is through *top-down* approaches, in which secondary porosity is introduced via post-synthetic modification of a pre-existing microporous matrix.^[1,2]

In this contribution, a *top-down* method was employed to obtain hierarchical porous materials with different properties. In detail: two commercial zeolites, HZSM5 and HUSY (both with SiO₂/Al₂O₃ = 80) with a MFI and FAU structure respectively, and a natural clinoptilolite (HCLI, Si/Al = 5) with HEU structure were desilicated using NaOH solutions at varying concentrations, to evaluate the treatment efficiency. The synthesized hierarchical architectures were characterized by multi-technique approach using XRD, N₂ physisorption at 77K, TGA and FTIR spectroscopy of adsorbed probe molecules, to assess the nature, strength and accessibility of the acid sites (Figure 1A,A').

The obtained hierarchical zeolites were evaluated for their ability to recover Cu²⁺ and Co²⁺ ions from aqueous solutions by using UV-Vis spectroscopy. Selectivity tests were also carried out to determine the materials' affinity for the target ions. The three investigated zeolites exhibited distinct behaviors, based on their different physicochemical properties (Figure 1B). HZSM5 demonstrated the highest adsorption capacity but showed no specific affinity for either metal ion, in contrast to HUSY and HCLI, which displayed a clear preference for Co²⁺ (Figure 1B). Notably, the desilication treatment significantly enhanced the adsorption efficiency of the studied materials.

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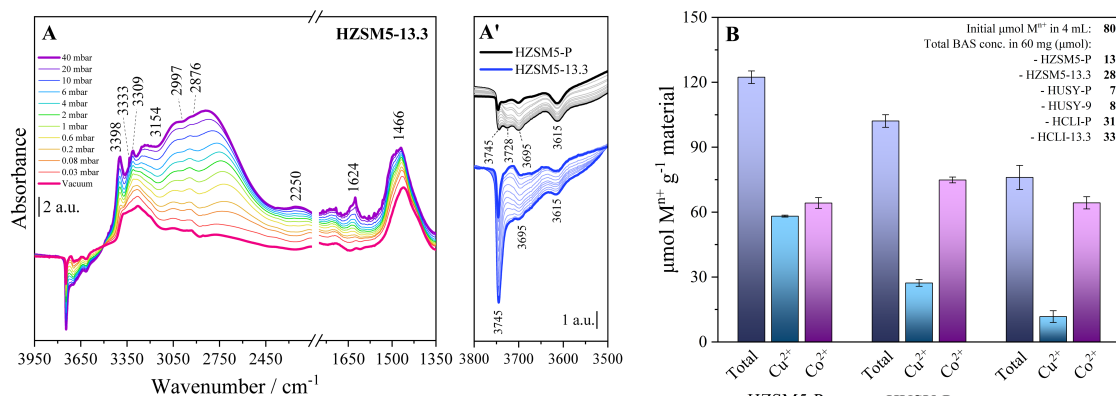


Figure 1: FTIR difference spectra of NH₃ adsorbed on hierarchical HZSM5 at room temperature (A); comparison of HZSM5 spectra before and after desilication in the OH stretching region (A'); Amount of Mⁿ⁺ captured by the studied zeolites from aqueous solution after 24 hours, expressed as μmol g⁻¹(B).

[1] M. Hartmann, Angew. Chem. Int. Ed. 43 (2004) 5880–5882.

[2] S. Mitchell et al. Nat. Commun. 6 (2015) 8633-8647.