Synthesis and Characterization of a Novel Zeolite, CJS-1

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A novel zeolite, CJS-1, has been synthesized from pip–Al_2O_3–SiO_2–HF–H_2O systems (pip = piperazine) and characterized by means of *X*-ray powder diffraction, scanning electron microscopy, thermal analysis, and gas adsorption.

Zeolites are usually obtained by hydrothermal crystallization from an alkaline reaction medium.[1,2] A new route to prepare pentasil zeolites was developed by Guth *et al.*,[3] using a non-alkaline medium in the presence of fluoride ions. In this way, the silica-MFI, Al-MFI, and Ti-MFI zeolites were obtained.[4] So far, there have been no reports on the synthesis of other zeolites in the non-alkaline medium. We report here the synthesis and characterization of a novel zeolite, CJS-1.

Crystallization of a reaction mixture with molar composition $0.5pip : 0.04Al_2O_3 : 1.0SiO_2 : 1.0HF : 30H_2O$ (pip =

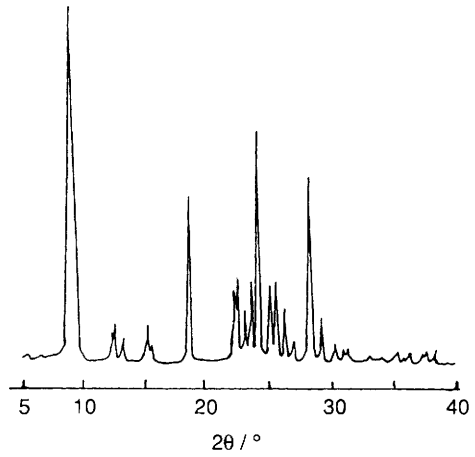

Figure 1. *X*-Ray powder diffraction pattern of CJS-1.

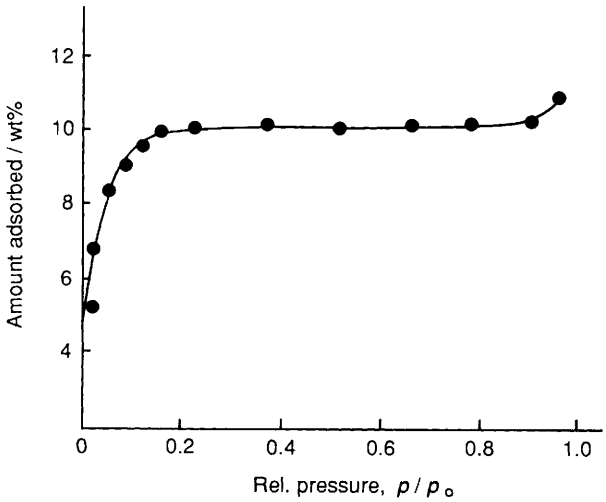

Figure 2. N_2 adsorption isotherm for CJS-1.

piperazine), was carried out in a Teflon-lined, 50 ml capacity autoclave at autogeneous pressure and 150 °C for 52 days. The silica and alumina sources were colloidal (SiO_2, 26%; Na_2O, 0.3%) and $Al_2(SO_4)_3 \cdot 18H_2O$ respectively. The crystalline product was filtered, washed with deionised water, and dried at ambient temperature. The *X*-ray powder diffraction pattern of CJS-1 is shown in Figure 1 (taken in the absence of the organic template, which was eliminated at 900 °C for 2 h). By comparison with the characteristic *X*-ray powder diffraction patterns of known zeolites, it was found that CJS-1 has a novel structure. Determination of the N_2 adsorption isotherm of CJS-1 (Figure 2) showed that it has characteristic micropore adsorption properties. The apparent surface area was 327 $m^2 g^{-1}$. The Langmuir type adsorption isotherms are similar to those of other zeolites.

Using differential thermal analysis–thermogravimetric analysis it was found that CJS-1 loses organic template at 510 °C and structurally collapses at 1122 °C. Scanning electron microscopy showed that the sample is pure in phase and highly crystalline. Polycrystalline morphology is observed, with an average size of 100 × 100 × 20 μm.

The molar ratio SiO_2/Al_2O_3 of CJS-1 was measured by chemical analysis to be 101.2. Elemental analysis indicated that CJS-1 has the empirical formula $0.1pip \cdot 0.01Al_2O_3 \cdot 1.0SiO_2$.

Thus, a novel zeolite, CJS-1, has been synthesized from pip–Al_2O_3–SiO_2–HF–H_2O systems. The structure consists of AlO_4 and SiO_4 as basic building units with an organic template. CJS-1 has regular intercrystalline pores.

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References

1 S. P. Zhdanov, 'Molecular Sieve Zeolites-1,' ACS, Washington, 1971, p. 20.
2 D. W. Breck, 'Zeolite Molecular Sieves, Structure, Chemistry and Uses,' Wiley, New York, 1974, p. 245.
3 J. L. Guth, H. Kessler, and R. Wey, in 'Proc. 7th Int. Conf. Zeolites,' eds. Y. Murakami, A. Iijima, and J. W. Ward, Kodansha, Tokyo, 1986, p. 121.
4 S. Qiu, W. Pang, and S. Yao, in 'Zeolites: Facts, Figures, Future,' eds. P. A. Jacobs and R. A. van Santen, Elsevier, Amsterdam, 1989, p. 133.