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Advances in high-resolution powder diffraction for applications in materials chemistry

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High-angular resolution powder diffraction, with well-defined and resolved diffraction peaks, is used generally to obtain high-quality data for complex structure refinements. The ID22 beamline at ESRF has very high angular resolution, arising from the combination of the highly collimated beam from the new EBS ring, wavelength selection via a cryogenically-cooled Si 111 monochromator, and use of a 13-crystal Si 111 multi-analyser stage between the sample and a Dectris Eiger2 X 2M-W CdTe pixel detector [1]. Such an arrangement, combining multi-analyzer stage and 2D detector, is the first of its type, allowing the low-angle asymmetry in the peak shape to be removed, angular resolution to be gained, and statistical quality of the high-angle data to be improved. Higher quality data can be collected faster, allowing complex in situ and operando experiments to be performed with applications in diverse areas of Materials Science. One of the applications is in relation with the current environmental challenges, to selectively trap green-house gases such as carbon dioxide (CO_2) and methane (CH_4) from flue gases (mix CO_2, H_2O, N_2) and biogas (mix CO_2, CH_4, H_2O) using microporous materials. For non-mixed gases, in situ adsorption experiments as a function of the gas pressure are carried out, and adsorption isotherms are retrieved after Rietveld refinement against the high-resolution powder diffraction data. Knowing the location of the gas molecules in the material allows a description of the adsorption process not only at the macroscopic scale, but also at the microscopic one, as each individual site is modelled with a site-specific isotherm with specific thermodynamic parameters. This deeper knowledge of the adsorption process opens the way to new strategies to optimize the selective removal of green-house gases from a competitive environment, as found in flue gases or biogas.

References

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