X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD for nanoscale sieve characterization

Application Report 39

X-ray diffraction is a key tool for characterizing the structure of ordered materials. Similar to the coherent scattering at higher angles (reflecting the arrangement of atoms), a coherent signal can also be observed at very low angles, reflecting the periodic arrangement of cylindrical pores in materials such as ZSM-41 or SBA-15.

Low angle data acquisition requires strict control of air scattering and of the illuminated area on the sample. While this is technically feasible with the proper selection of a fixed aperture primary divergence slit and a static anti-scatter screen, the use of Dynamic Beam Optimization (DBO), available on the D6 PHASER, makes such challenging experiments very convenient and straightforward. DBO combines the simultaneous use of a motorized divergent slit, a motorized air-scatter screen, and a variable detector aperture.

The D6 PHASER not only provides the arrangement and spacing of the pores. The pores can be used as host matrices into which nano-entities can be loaded, opening up a wide range of applications for SBA-15 and other mesoporous materials, such as catalysis, environmental treatment for adsorption and separation, drug delivery systems or gas sensors. The D6 PHASER also helps to ensure the integrity of the mesoporous structure after loading and to monitor any change in the spacing between the pores.

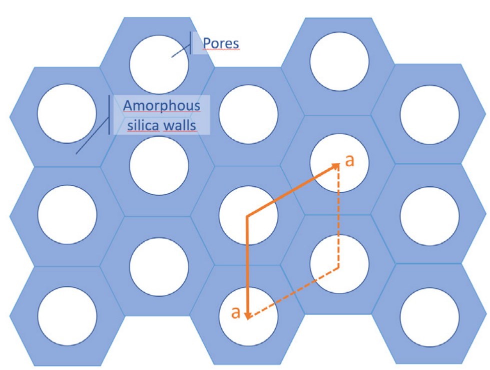
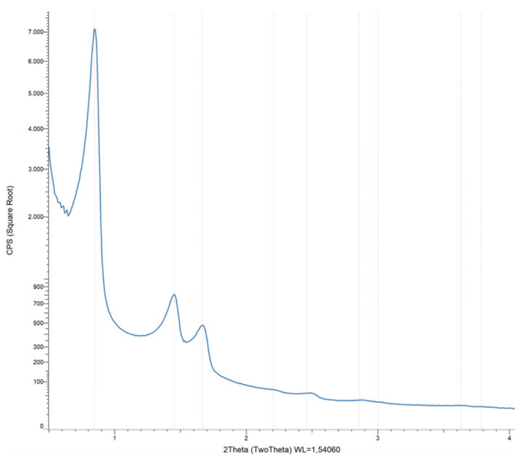


Figure 1
Structure of Santa Barbara Amorphous 15 (SBA-15), a mesoporous silica compound consisting of a short-range ordered silica matrix (light blue) and an ordered hexagonal array of cylindrical nanopores (white) with pore distance a.

SBA-15 is a mesoporous silica screen based on a framework of uniform hexagonal oriented pores. SBA-15 has a high internal surface area, typically 400-900 m^2/g, with a narrow pore size distribution and a tunable pore diameter of about 5 to 15 nm and relatively thick walls ranging from 3.1 nm to 6.4 nm.

The scan in Figure 3 shows a pattern collected from 0.5° to 4° 2Theta where several peaks can be identified. The peaks can be indexed by a two-dimensional hexagonal symmetry. The first peak is observed at an angle as low as 0.82° 2Theta. A space group reflecting the hexagonal symmetry (P6mm) is used to refine the data (Figure 4). The fit yields a lattice parameter of a=12.5 nm in the hexagonal plane, corresponding to the pore-to-pore distance.

Figure 2
D6 PHASER equipped for Dynamic Beam Optimization (DBO) with Variable Divergence Slit (VDS), Motorized Air Scatter Screen (MASS) and LYNXEYE XE-T detector.



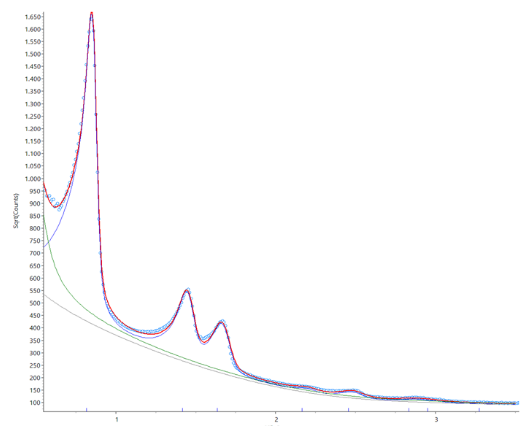


Figure 3
SBA-15 Low-angle diffraction peaks.

Figure 4
SBA-15 Pawley fit using DIFFRAC.TOPAS.

Data were collected with a D6 PHASER 1.200 W, Cu radiation, no filter, 1.5° Soller collimators, primary variable divergence slit and motorized air-scatter screen, and LYNXEYE XE-T detector closed to 0.3° active range. Total scan time was 21 min.

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