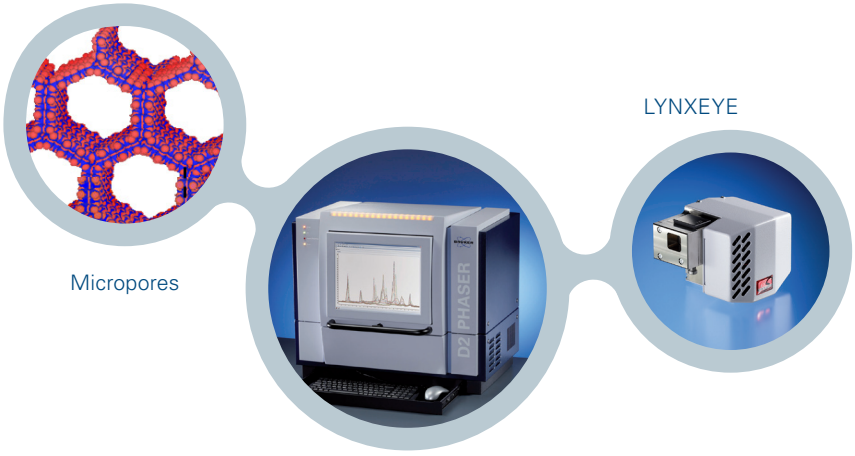


Application Report XRD 13

D2 PHASER Desktop XRD: SAXS-Analysis of the Mesoscopic Catalyst SBA-15

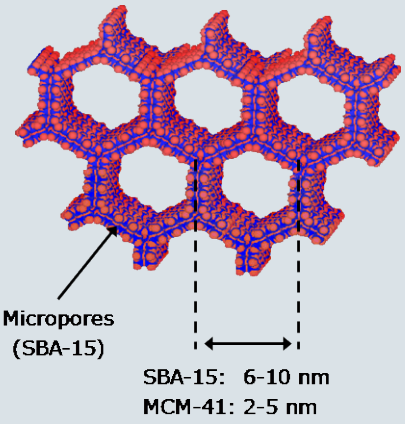
The D2 PHASER is a portable desktop XRD instrument for research and quality control. It is easy to operate and independent of external media such as cooling circuits. Thanks to the LYNXEYE detector it is the fastest desktop XRD system on the market. This report demonstrates its use for fast and reliable SAXS measurements of material exhibiting large d-spacings up to about 10 nm.

Catalysts are indispensable to modern-day society because of their prominent role in petroleum refining, bulk and fine chemical processing and reduction of environmental pollution. High surface-to-volume ratios are often important for these particles since catalytic processes take place at the surface. Therefore, supports such as silica and gamma-alumina are generally used to obtain small and thermally stable particles.

D2 PHASER, LYNXEYE detector
Cu radiation (30 kV, 10 mA), Ni filter
Continuous scan from 0.6 to 10.0° 2Theta, Step width 0.01° Counting time 0.2 sec per step
Total scan time about 4 min.
2.5° Soller collimators, 0.1 mm divergence slit, secondary anti-scatter slit 3mm, air-scatter screen over sample closed to 0.1 - 0.2 mm
LYNXEYE detector opening 1° 2Theta

Fundamental studies are often hampered by the heterogeneity of conventional supports that make it difficult to disentangle the effects of the individual preparation steps on the final dispersion. To overcome these problems meso-porous silica SBA-15 (Santa Barbara no. 15) can be used as a support system. Figure 1 (left) shows the structure, which essentially consists of an amorphous silica framework forming a two-dimensional hexagonal primitive assembly of straight channels or pores. The structure itself is flexible and may adopt different pore diameters. The pore size can be measured from TEM pictures as shown in Figure 1 (right).

X-rays play an important role in the characterization of these materials. Figure 2 shows small-angle powder data of CuO loaded SBA-15. The three peaks labeled in graph 2 are caused by the regular array of the pores. They are a measure of the average pore distance.



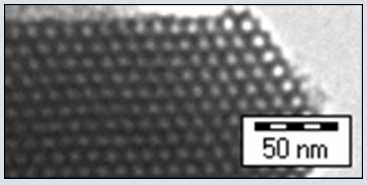


Figure 1: Schematic view of zeolite SBA-15 (left). This material exhibits pores up to 10 nm diameter which is larger than for other meso-porous materials such as MCM-41. The size of the pores is also seen on TEM picture (right).

A major advantage of powder X-ray scattering over other methods is the negligible effort needed to prepare the sample. The measurement is very fast and takes a few minutes only. Moreover, XRD has a superior sensitivity to dimensional changes of material on the below 10 nm length scale.

The example presented in Figure 2 indicates that impregnation of SBA-15 with CuO during the preparation of the catalyst does not affect the long-range order of the pores in the support material.

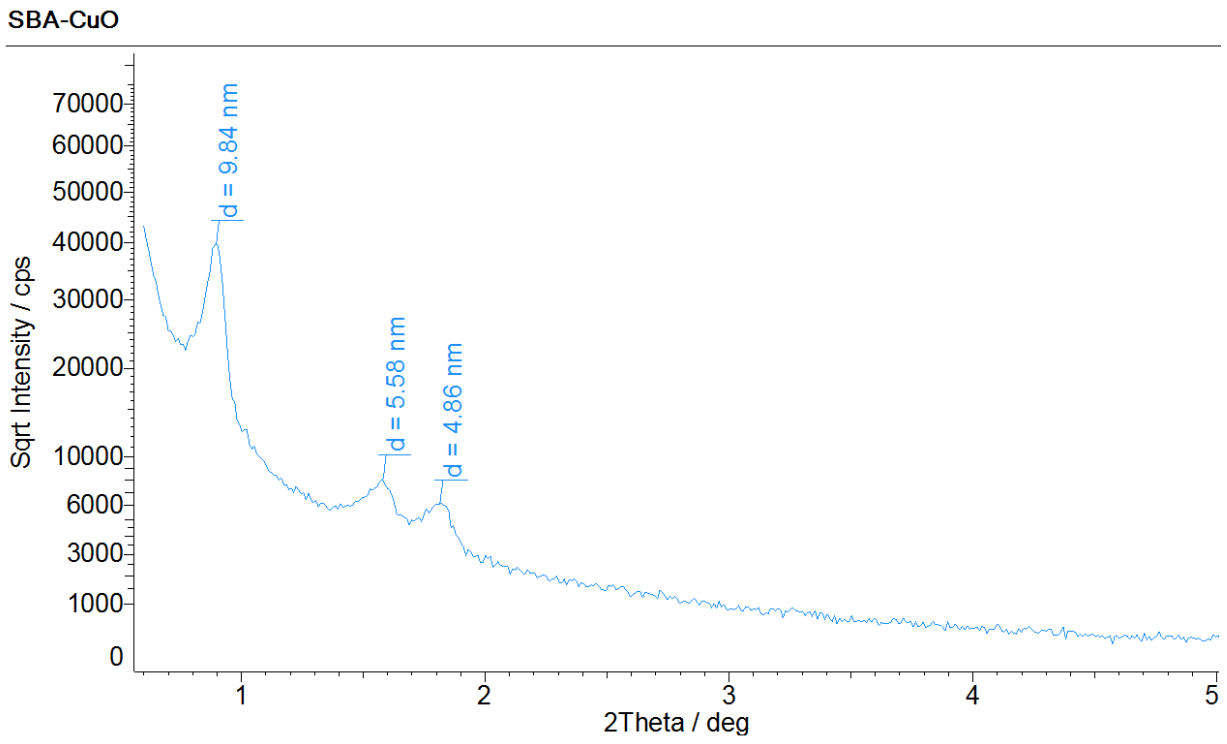


Figure 2: Small-angle X-ray powder pattern of CuO loaded SBA-15. The three signals correspond to the 100, 110 and 200 reflections on a hexagonal primitive lattice. They are used to estimate the average pore distance of this material, here about 11.25 nm.

Sample and graphics are courtesy of the Inorganic Chemistry and Catalysis group, Department of Chemistry, Utrecht University, The Netherlands.

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