crystals

MDPI

Supplementary Material

Quantum-Sized Zinc Oxide Nanoparticles Synthesised within Mesoporous Silica (SBA-11) by Humid Thermal Decomposition of Zinc Acetate

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Characterization method

Powder X-ray diffraction (XRD) measurements were performed using a Bruker AXS D8 ADVANCE diffractometer with a copper target (λ = 1.5418 Å). The DIFFRACplus software was used for analysis, and the following operational parameters were employed: 40 kV, 40 mA, front slit window size 0.1 mm for low angle, 1.0 mm for wide angle scans. The deflection plate was placed at a distance of 0.5–1.0 mm above the sample for the low angle scan; continuous coupled two-theta scan/theta scan mode, 0.5 step/s. Nitrogen gas adsorption–desorption measurements were performed using a Micrometrics Tristar analyser. Surface areas were produced by Brunauer-Emmett-Teller (BET) method, whereas average pore sizes were obtained from a Barrett-Joyner-Halenda (BJH) adsorption cycle. Transmission electron microscopy (TEM) imaging was performed using a JEOL JEM-3011 electron microscope operating at 250 kV. The images were recorded using a Gatan 794 CCD camera. Gatan Digital Micrograph software was used to analyze the TEM images. The TEM instrument was equipped with a PGT prism Si(Li) energy dispersive X-ray spectroscopy (EDX) detector and Avalon 2000 analyzer, which was used for determining the chemical compositions of the samples. EDX was performed in TEM mode using a 7 nm beam diameter for an acquisition time of 1 min. X-ray photoelectron spectroscopy (XPS) measurements were performed using a Thermo ESCALAB 250Xi spectrometer with a monochromator and an Al-Kα radiation source (1486.6 eV). The spectra were recorded and processed using an Avantage data system. The analyses were carried out using the following parameters: analysis chamber pressure of 10–9 Torr, step size of 0.1 eV, dwell time of 100 ms, and pass energy of 20 eV. All binding energy values were determined with respect to the C1s line (284.6 eV) originating from adventitious carbon. A flood gun was used in the standard charge compensation mode to neutralize the charge build-up on the surface of the insulating layer. The UV-Vis spectroscopy was performed using a Cary 5000 UV-Vis-NIR spectrophotometer (Version 1.12) with the following settings: abs mode 200–800 nm, scan rate 600.000 nm min^{-1}, data interval 1.000 nm, full slit height, double beam mode, signal to noise mode off, and baseline correction on. The powder samples were pressed into a disc shape without dilution and an SBA-11 disc was used as reference sample for the UV-Vis spectroscopy.

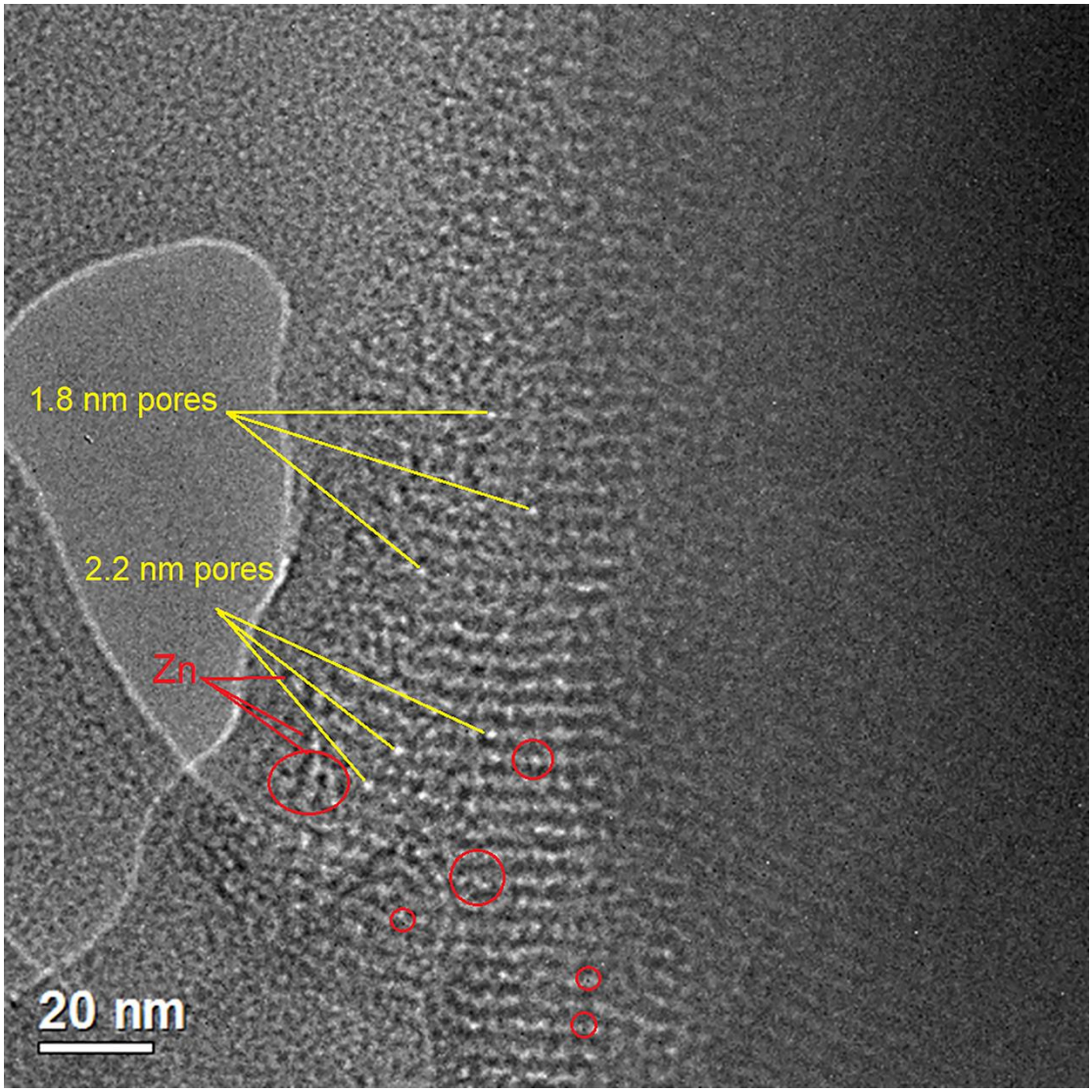

Figure S1. Bright field TEM image of ZnO-SBA-11. A lower magnification image is shown in Fig. 2(a).

The BF TEM image in Fig. S1 is a high magnification image of Fig. 2(a) presented in the main paper, which clearly shows some of the exposed pores have sizes of 1.8 to 2.2 nm as indicated by the yellow lines. Bearing in mind that some of these pores are blocked by the top or bottom layers of the sample because the electron beam can only penetrate through a few layers and the 3D structure is projected into a 2D image. Therefore, pore sizes might appear smaller than they are, especially for this cubic 3D structure at which the pores are interlocking. A more accurate pore size measurement was obtained from the N_2 sorption analysis. There are a number of dark spots in the bright field TEM image (Fig. S1), some of which are indicated by the red lines and circles. These might indicate the Zn atoms, as the atomic weight of Zn is 65 g/mol compared to 28 g/mol for Si and therefore Zn-rich regions should appear darker than Si in bright field TEM images.