Supporting Information

Size-dependent CO_2 capture in chemically synthesized magnesium oxide nanocrystals†

Anne M. Ruminski,[1] Ki-Joon Jeon,[2‡] and Jeffrey J. Urban[1]

[1]The Molecular Foundry, Material Science Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720. [2]Environmental Energy Technologies Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720. [‡]Present address: School of Electrical Engineering, University of Ulsan, Ulsan 680-749, Republic of Korea E-mail: jjurban@lbl.gov.

† This paper is part of a Journal of Materials Chemistry theme issue on the chemical transformations of nanoparticles.

Materials and Characterization: MgO -325 mesh (44 μm, MgO-325M) and MgO 40 mesh (420 μm, MgO-40M) were purchased from Strem Chemicals and Acros Organics, respectively. The size and morphology of the MgO nanoparticles (MgO-NP1 and MgO-NP2) were analyzed by transmission electron microscopy (JEOL 2100F TEM, 200 kV accelerating voltage). For imaging, MgO NPs were dispersed in toluene then a drop of the suspension was deposited onto a TEM grid (Ted Pella, lacey carbon and ultra thin carbon 300 mesh Cu grid). The size and morphology of commercially purchased MgO (MgO-325M and MgO-40M) was examined by scanning electron microscopy (Zeiss

Ultra 55 Field Emission SEM). X-ray diffraction patterns were obtained with a Bruker D8 Discover X-ray diffractometer with a general area detector diffraction system (GADDS) using $Cu_{K\alpha}$ radiation (λ = 0.154 nm). For Debye-Scherrer peak analysis of XRD spectra to calculate nanocrystal diameters, a Scherrer constant of 0.9 was used for spherical MgO-NP1 while a constant of 1.3 for disk-like MgO-NP2. Infrared spectra were recorded on a PerkinElmer FT-IR Spectrum One spectrophotometer. Spectra of chemically synthesized MgO nanocrystals were acquired by drop casting a toluene solution containing MgO NPs on a attenuated total reflectance FTIR attachment. Spectra of heat-treated MgO samples were acquired by transmission FTIR. Samples were made into a potassium bromide (KBr) pellet by mixing 1 mg MgO sample with 100 mg KBr followed by subjection to high pressure using a manual pellet maker. Thermogravimetric analysis was performed under N_2(g) at a scan rate of 5 °C /min using a TA instruments Q5000 TGA. N_2 adsorption/desorption measurements were acquired with a Micromeritics TriStar II 3020 surface area and porosity system.

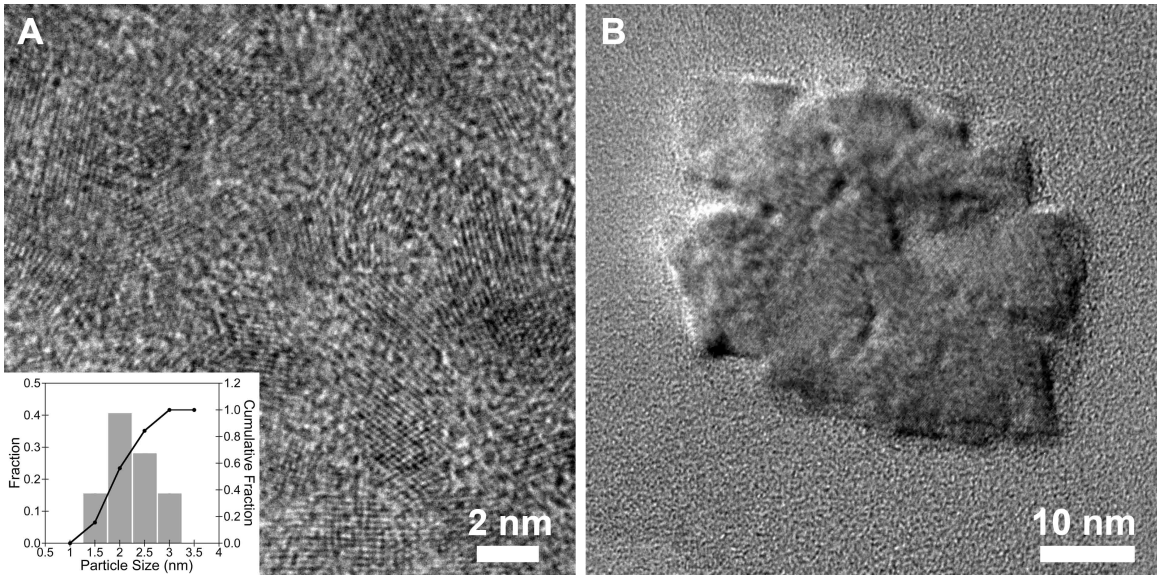

Figure S1. Transmission electron microscope images of as synthesized: (A) MgO-NP1, mean particle diameter is 2.5 ± 0.5 nm. (B) MgO-NP2, with particle width ranging between 32 and 53 nm.

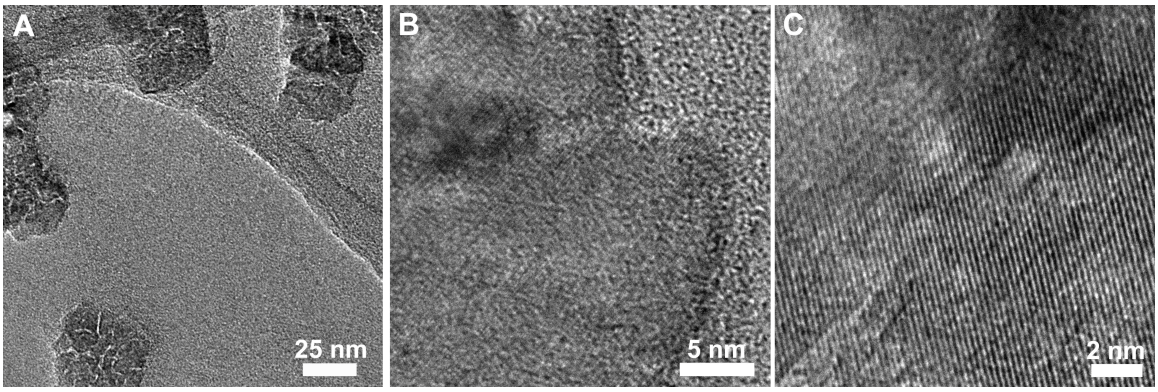

Figure S2. TEM images of as synthesized MgO-NP2. (A) Low-resolution of multiple particles, demonstrating the disk-like morphology. (B) Image taken at the nanodisk edge. The contrast is low revealing the carbon grid below the sample, signifying that the sample is very thin and thus indicating that MgO-NP2 is disk shape instead of spheroid like MgO-NP1. (B) High-resolution image reveals that these particles are crystalline, and intensity variations indicate that these samples contain several grain boundaries.

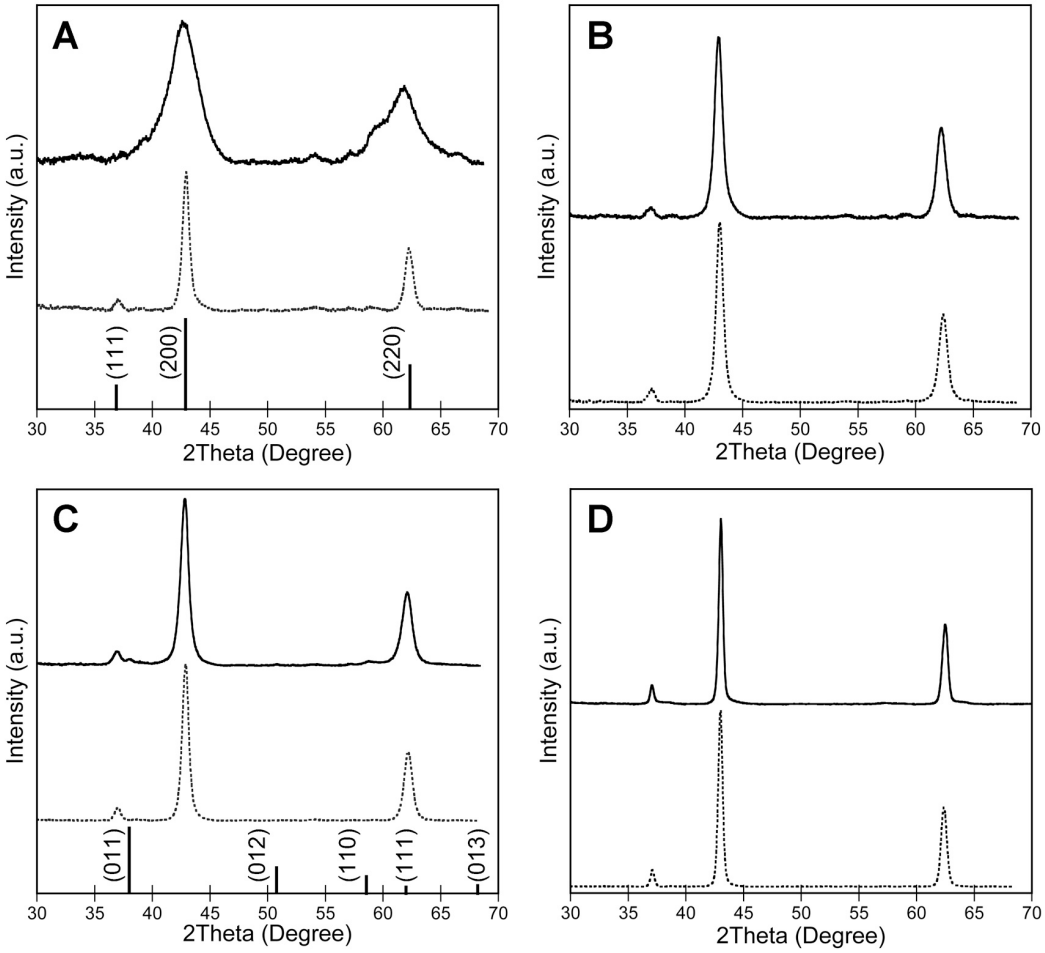

Figure S3. X-ray diffraction patterns of MgO particles before (solid line) and after (dotted line) heat treatment at 600 C: (A) MgO-NP1, (B) MgO-NP2, (C) MgO-325M and MgO-40M. Reference diffraction pattern of cubic MgO (JCPDS 07-0239) is included in bottom of panel (A), and hexagonal $Mg(OH)_2$ (JCPDS 01-083-0114) in bottom of panel (C). Weak peaks from $Mg(OH)_2$ are visible in the spectrum of MgO-325M at 38.0, 50.8 and 58.6 degrees before heat treatment. Debye-Scherrer peak analysis of the nanocrystal spectra before and after heat treatment calculated diameters of 2.7 ± 0.2 nm and 9 ± 3 nm for MgO-NP1 pre and post heat treatment, respectively. Analysis of MgO-NP2 spectra produced diameters of 15 ± 2 nm and 16.6 ± 0.7 nm pre and post heat treatment, respectively.

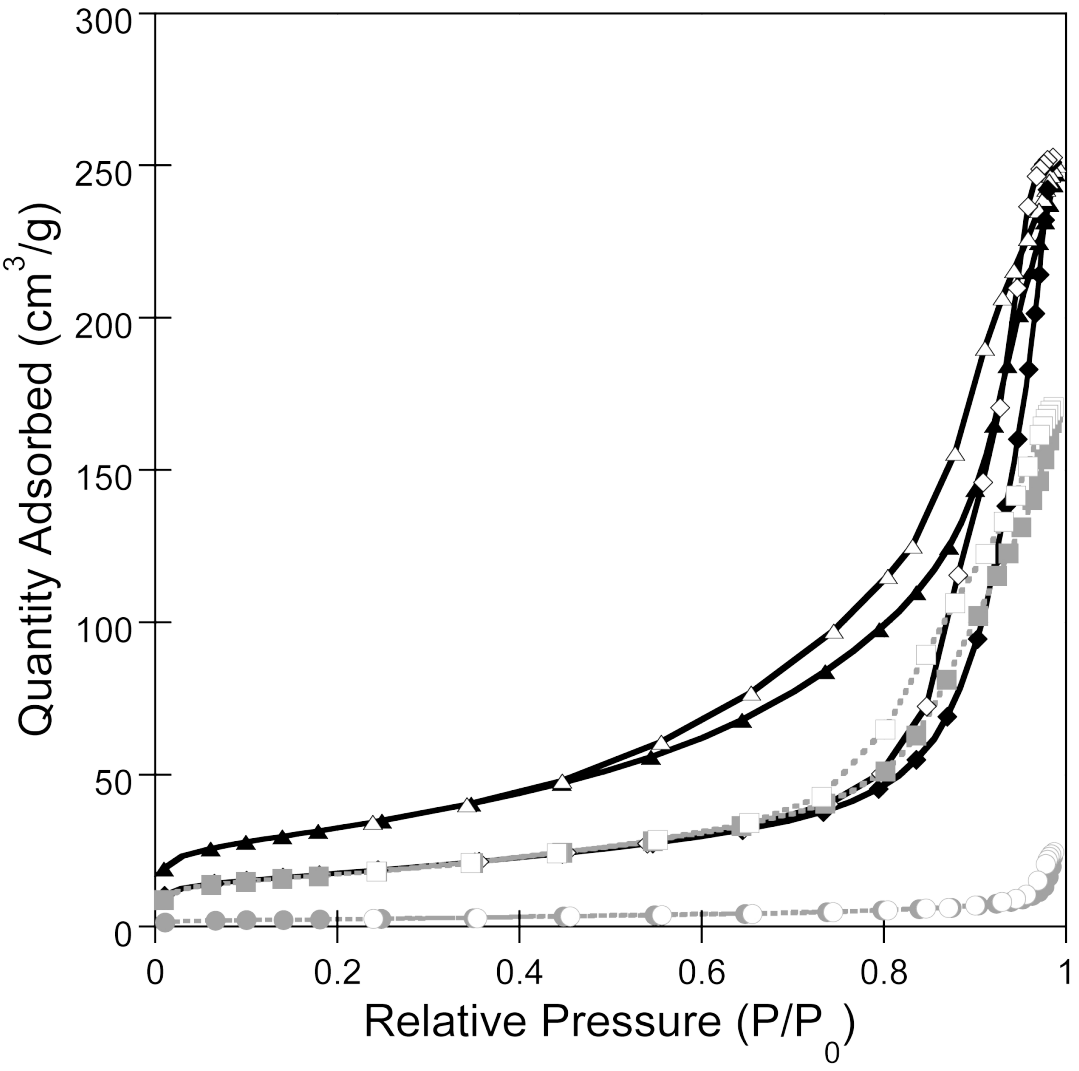

Figure S4. Overlaid N_2 adsorption (solid symbols) and desorption (open symbols) graph of the four heat-treated MgO samples: MgO-NP1 (5 nm, black triangles), MgO-NP2 (23 nm, black diamonds), MgO-325M (44 μm, grey squares) and MgO-40M (420 μm, grey circles).

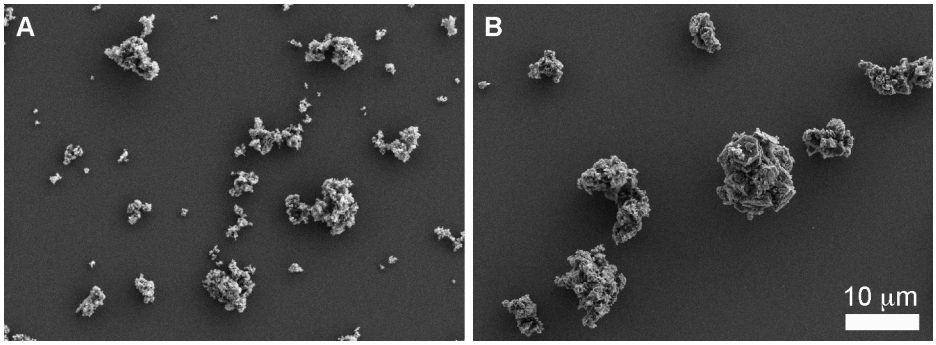

Figure S5. Scanning electron microscope images of preconditioned heat-treated (A) MgO-325M and (B) MgO-40M.

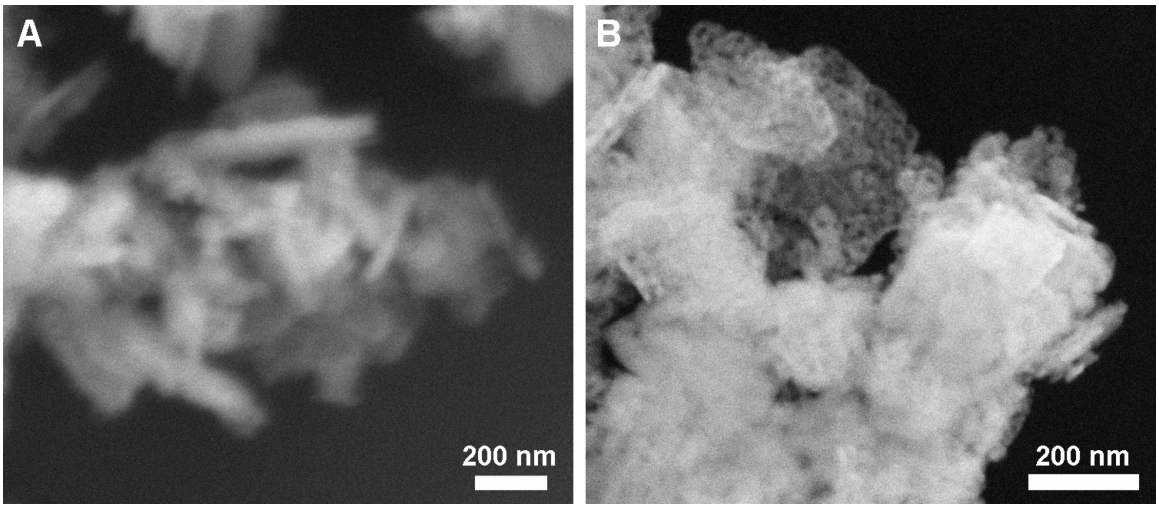

Figure S6. Scanning electron microscope images of MgO-325M after (A) preconditioning heat treatment and (B) an extended temperature exposure of 600 °C under nitrogen for 15 hours.