Supplementary Material (ESI) for Chemical Communications
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One-minute synthesis of crystalline binary and ternary metal oxide nanoparticles

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Electronic Supplementary Information

Chemicals:
Mn(II) acetate 98%, Mn(II) acetylacetonate, Zn(II) acetylacetonate hydrate 99.995%, Fe(II) acetate 99.995%, Fe(II) acetylacetonate 99.95%, Fe(III) acetylacetonate 99.9+%, Co(II) acetate 99.995%, Ba metal 99.99%, and $Ti(OiPr)_4$ 99.999% are used as precursors and anhydrous benzyl alcohol 99.8% as solvent. All the chemicals were purchased from Sigma-Aldrich and used as received.

Synthesis:
In general, 1 Mmol of the metal oxide precursor was dissolved in 5 ml of benzyl alcohol under an inert atmosphere and transferred into a glass tube, sealed with a Teflon cap, and taken out of the glovebox. In the case of $BaTiO_3$, 3.5 Mmol Ba were dissolved in 5 ml benzyl alcohol at 50 °C, followed by the addition of 1 mol equivalent $Ti(OiPr)_4$. The reaction mixture was heated to 200°C. After the thermal treatment, the precipitate was extracted by centrifugation and each sample was washed with ethanol and diethyl ether. The wet solid samples were dried in an oven at 60 °C overnight. The powders obtained were ground in a mortar. In the case of cobalt oxide, two sets of five solutions of 0.1 M and 0.2 M $Co(acetate)_2$ in benzyl alcohol were prepared. The solutions were heated to 200°C and the irradiation time was changed in the range of 30 seconds to 20 min. Reaction yields were calculated on the basis of the theoretical weight of CoO with respect to the starting concentration of the cobalt acetate. For the study of the reaction mechanism the precipitates were separated from the reaction solution, which was kept for GC-MS.

Microwave heating protocol (Figure ESI-1):
During a typical run, the power is adjusted to heat the sample up to 60°C, and then the temperature is kept for one minute with high stirring rate to allow a good dispersion of the precursor in benzyl alcohol. The organic reaction is induced by increasing the power to 300 W and rapidly heating the solution to 200 °C (see Figure ESI-1). The temperature is kept constant during the different reaction times and then quenched by a compressed air flow. The reaction is performed in sealed vessels (inner volume of 10 ml) with magnetic stirring and under microwave irradiation. The temperature and the pressure are controlled by an IR thermometer and a pressure sensor, respectively.

Instruments and Characterization:
The microwave experiments were conducted using a CEM Discover reactor operating at a frequency of 2.45GHz. The basic configuration of the CEM microwave furnace system consists of a microwave source (magnetron), control device and a single mode resonant cavity. The cavity is equipped with a variable speed magnetic stirring mechanism, an IR temperature and a pressure sensor. The power is modulated in function of the temperature and has a maximum operating power at 300 W. A compressed air flow allows the fast cooling of the sample. X-ray diffraction (XRD) were performed with a Panalytical diffractometer using Cu Kα λ=1.5406 Å radiation

and a STOE powder diffraction system using Co Kα, λ=1.78892 Å radiation. The particle size was determined by the Scherrer equation, assuming that the nanoparticles were spherical. In the time dependent experiment the sample loading and measurement conditions were the same for all the samples.

Transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM) measurements were performed on a CM30ST microscope (Philips; LaB6 cathode, operated at 300 kV, point resolution of 2 Å). The samples were ground and then suspended in ethanol. One drop of this suspension was placed on a 400-mesh carbon-coated copper grid and left in air to dry. To prevent agglomeration of the nanoparticles the copper grid was placed on a filter paper. Gas-chromatography coupled with mass spectrometry (GC-MS) was performed on a Trace MS, Thermo Quest (quadrupol MS) with a Zebron ZB-1ms (Phenomenex) column [length 60 m, inner diameter 0.25 mm, stationary phase ZB-1 (dimethylpolysiloxan), film thickness 0.25 µm, mobile phase: helium with a flow rate of 0.8 mL/min, split ratio of 1:500]. The heating program was isothermal 5 min at 40 °C; heating with 20°C/min up to 250 °C. MS: mass acquisition m/z 10 - 300; scan rate 2.5 scans/s.

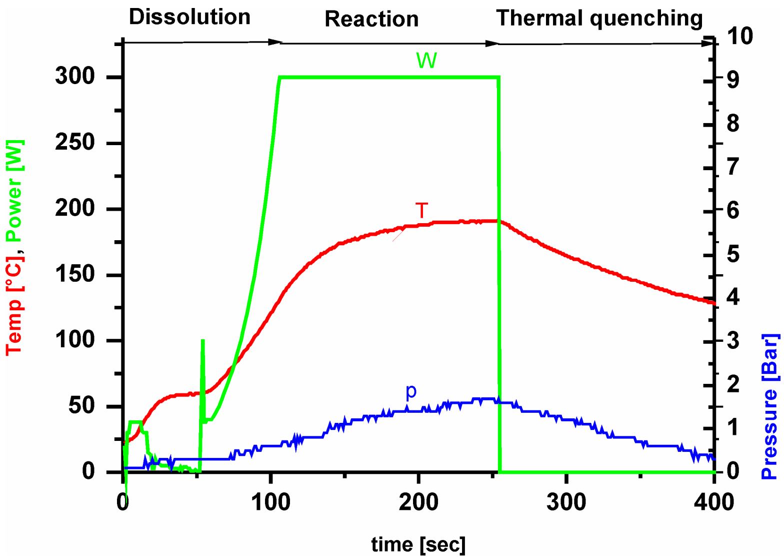

Figure ESI-1. Typical temperature, pressure and power profile used for the synthesis of the metal oxide nanoparticles.

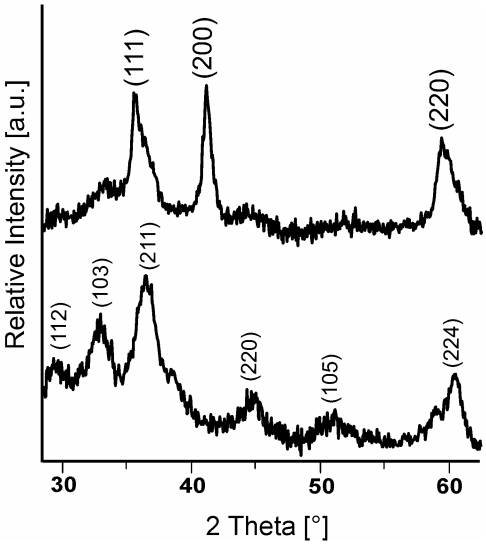

Figure ESI-2. XRD powder pattern of MnO (upper pattern), and Mn_3O_4 (lower pattern) obtained using Cu Kα.

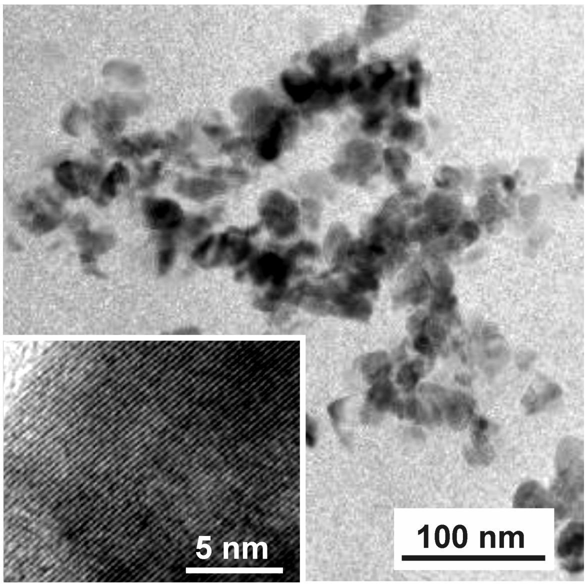

Figure ESI-3. TEM image of ZnO nanoparticles (inset: HRTEM image of a part of one nanoparticle).