

Supporting Information

Nano-MgO-ZrO₂ Mixed Metal oxides: Characterization by SIMS and application in the reduction of carbonyl compounds and in multicomponent reactions

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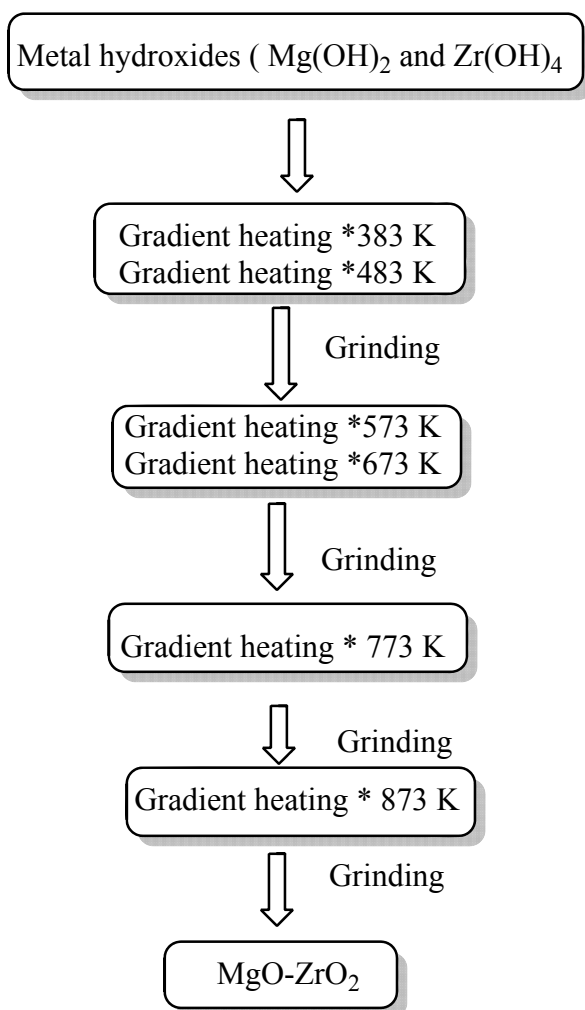
Experimental

All commercial reagents were used as received unless otherwise mentioned. For analytical and preparative thin-layer chromatography, Merck, 0.2 mm and 0.5 mm Kieselgel GF 254 percoated were used, respectively.

Synthesis of Nano-MgO-ZrO₂

In continuation of our interest to explore the utility of MgO-ZrO₂ catalyst in organic synthesis, we have successfully prepared MgO-ZrO₂ catalyst, by Ultra dilution method as described below.

In a typical experiment, for the preparation of MgO-ZrO₂ an appropriate amount of magnesium nitrate [Mg(NO₃)₂.6H₂O] (3.10 g) and zirconium oxychloride [ZrOCl₂.8H₂O] (8.11 g) were dissolved together in 2 L flask with 1 L deionized water. Dilute ammonia solution was added drop wise with vigorous stirring (RPM- 5,000) until the precipitation was complete (around 6 to 8 h and pH= 10.0). The resultant precipitate was filtered and washed with distilled water till free from chloride ions. The residue was dried for 24 h at 383 K in an oven and the obtained precipitate of metal hydroxides heated in porcelain crucible progressively to 873 K for 10 h.



Catalyst characterization

After calcinations, the catalyst was characterized by various analytical and spectroscopic techniques. The X-ray powder diffraction pattern was obtained using a conventional powder diffractometer (Philips 1050) using graphite monochromatized Cu-K α radiation operating in Bragg-Brentano ($\theta/2\theta$) geometry. Transmission Electron Microscopy (TEM) experiments were performed on a Hitachi 8100 microscope with Rontec standard EDS detector and digital image acquisition. All samples were prepared by evaporating dilute suspensions on carbon-coated film. To produce a plain and conductive sample suitable for SIMS analysis, the powder catalyst was pressed onto an ultra-pure indium foil by Goodfellow (Huntingdon, UK). We used a manual toggle pressing machine by Brauer (Milton Keynes, UK). TOF-SIMS analysis was performed by acquiring positive and negative secondary ion spectra in the mass range of 0.5-200 m/z with an upgraded VG Ionex IX23LS TOF-SIMS set-up based on the Poschenrieder design.²⁸⁻²⁹ A focused liquid Ga⁺ gun in the pulsed mode (6 kHz/ 40 ns) was used as a

source of the analysis ions. A beam current in the continuous mode at 14 keV was ca. 15 nA with a raster size of $300 \times 300 \mu\text{m}^2$ (128×128 pixels, 10 kHz). The sample potential was ± 5 kV. Vacuum during the experiments was maintained in the range of $(2-3) \times 10^{-9}$ mbar in the analytical chamber.

TEM of MgO-ZrO₂ –

The TEM Image of MgO-ZrO₂ is depicted below, clearly indicates the particles are in the Nano size range (20 nm -35 nm) (Figure 1).

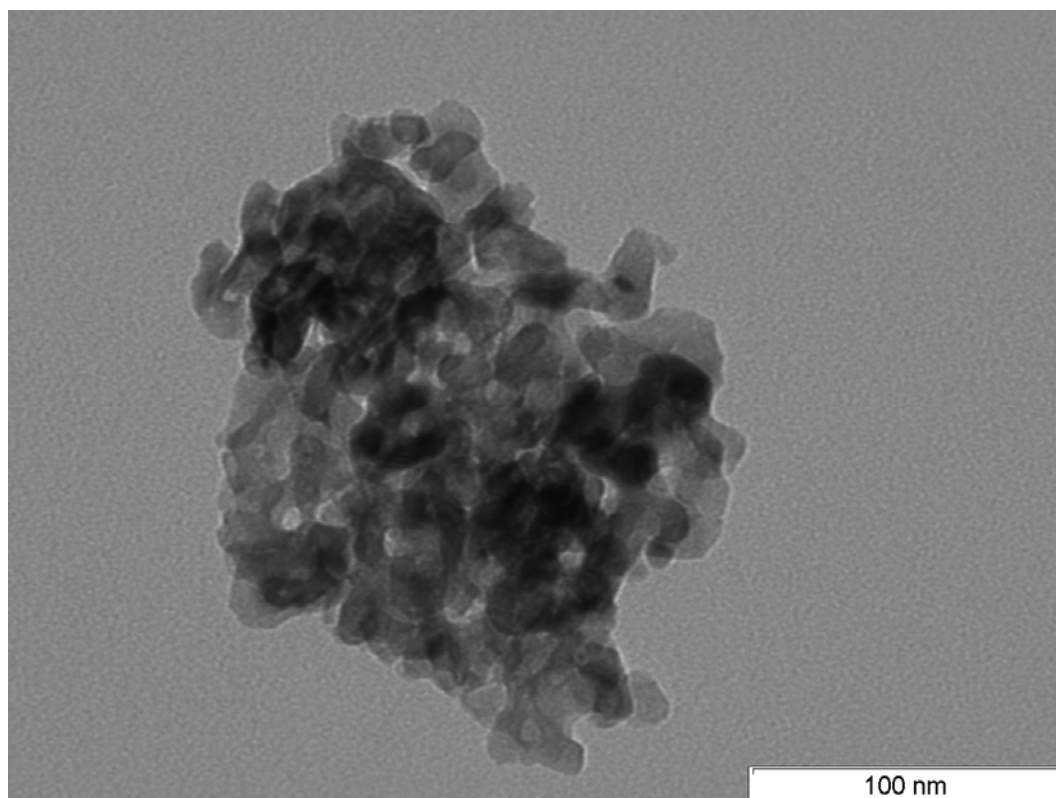


Fig. 1 TEM image of MgO-ZrO₂ at 100 nm