Preparation and characterization of the nanostructured cerium dioxide sorbents

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AIM: Preparation and characterization of CeO₂ nano-sorbents used for the removal of pesticides and nervous agents.

Sample preparation
- An input material for preparation of samples was cerium(III) nitrate, Ce(NO₃)₃·6H₂O, dissolved in deionized water.
- An insoluble cerous carbonate precipitated using NH₄HCO₃ (precursor 1) and using homogeneous hydrolysis of urea (precursor 2).
- Cerium(III) carbonate was filtered, washed with deionized water number of times and dried at 110 °C in a box drying apparatus for several hours to remove excess water and to form CeO₂.
- The temperature treatments of the precursors were done in an open porcelain crucibles at temperatures ranging between 500 °C/2 h and 800 °C/2h, step 100 °C in muffle furnace.

Experimental techniques
- SEM (Scanning Electron Microscopy) – TESCAN LYRA 3XMU FEG/SEM microscope with an Xmax 80. Oxford Instruments detector for EDX
- XRPD (X-Ray Powder Diffraction) – XPert PRO diffractometer by PANALYTICAL, CoKα irradiation (λ = 0.1789 nm), Bragg-Brentano geometry, 2θ range 20° ± 135°
- MS (Mössbauer Spectroscopy) – at room temperature in transmission geometry using a ⁵⁷Co/Rh source, velocity calibration performed by α-Fe, data evaluated using the program CONFIT
- SQUID (Superconducting Quantum Interference Device) – Quantum Design MPMS5, magnetization curves measured at room (300 K) and low (2 K) temperatures with maximal applied magnetic field ± 70 kOe (7 T), field-cooled (FC)/zero-field-cooled (ZFC) curves

Precipitation using NH₄HCO₃

Microstructure

Homogeneous hydrolysis

\( \sigma \) – lattice parameter, \( \delta \) – crystallite size

Magnetic properties
- Both precursors show linear dependence of magnetization on the applied external magnetic field.
- Hysteresis loops of annealed samples exhibit ferromagnetic response at low magnetic fields followed by a para- and diamagnetic behaviour at higher fields.
- Saturation magnetization of annealed powders is very low (thousandths of emu/g) without clear dependence on the annealing temperature.
- As confirmed by Mössbauer spectroscopy the ferromagnetic response comes from the low amount of iron oxide nanoparticles (units of ppm).
- Hyperbolic shapes of the FC/ZFC curves in the temperature range 2 K – 300 K at constant magnetic field 1 T indicate paramagnetic behaviour of CeO₂ with the Curie constant about 3 \( \times \) \( 10^{-7} \) (emu K)/g Oe.

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