AIM: Microstructural and magnetic characterization of magnetically separable CeO₂/Fe-oxide powder sorbents applicable towards selected pesticides. Analysis of magnetite transformation in dependence on calcination temperature and CeO₂ content.

### SAMPLE PREPARATION

- **Magnetically separable sorbent** – composite material consisting of iron oxide serving as a magnetically separable core or carrier and cerium dioxide (CeO₂) serving as active constituent capable to destroy dangerous chemicals.
- **Magnetite core** – synthesized by the co-precipitation of the Fe³⁺ (ferrous sulphate monohydrate) and Fe⁴⁺ (ferric sulphate) salts from cheap and commercially available raw materials.
- **CeO₂/Fe-oxide reactive sorbents** –
  - ferromagnetic core re-dispersed in the solution containing cerium (III) nitrate, and the cerium (III) carbonate prepared by precipitation with ammonium hydroxide carbonate
  - cerus carbonate/magnetite precursor annealed in a muffle furnace at various temperatures T_f from 473 to 1073 K for 2h
  - two series of sorbents with 5 and 38 wt.% CeO₂ annealed at different temperatures

### EXPERIMENTAL TECHNIQUES

- **XRD (X-Ray Diffraction)** – XPERT PRO diffractrometer (Panalytical) equipped with Co Kα radiation (λ = 0.17902 nm), 2θ range 20° - 135°, – evaluation – Rietveld structure refinement method by using the HighScore Plus program and the ICSD database
- **SEM (Scanning Electron Microscopy)** – TESCAN LYRA 3XMU FEG/SEM, accelerating voltage 20 kV, equipped with an X-Max80 Oxford Instruments energy-dispersive X-ray (EDX) detector
- **VSM (Vibrating-Sample Magnetometer)** – Microsense EZ2, room temperature (RT) magnetization and virgin curves • maximal magnetic field 1600 kA/m, first-order reversal curves (FORC) with step 8 kA/m
- **PPMS (Physical Property Measurement System)** – Quantum design, Inc., field-cooled (FC) and zero-field-cooled (ZFC) curves in the temperature range 2-293 K in magnetic field of 8 kA/m

### XRD

The results of Rietveld analysis of sorbents calcined at T_f temperature; phase content (A), lattice parameters (a, b, c), microdomain size (d):

<table>
<thead>
<tr>
<th>T_f (K)</th>
<th>CeO₂</th>
<th>Fe₂O₃</th>
<th>Fe₃O₄</th>
<th>CeO₂</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>T_f (°C)</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>473</td>
<td>5.4</td>
<td>8.55</td>
<td>0.3</td>
<td>0.3</td>
<td>49.9</td>
<td>8.93</td>
<td>0.98</td>
<td>0.95</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>723</td>
<td>4.3</td>
<td>10.4</td>
<td>0.3</td>
<td>0.3</td>
<td>56.4</td>
<td>8.93</td>
<td>0.98</td>
<td>0.95</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>873</td>
<td>1.3</td>
<td>14.1</td>
<td>0.3</td>
<td>0.3</td>
<td>62.1</td>
<td>8.93</td>
<td>0.98</td>
<td>0.95</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1073</td>
<td>0.5</td>
<td>15.6</td>
<td>0.3</td>
<td>0.3</td>
<td>68.7</td>
<td>8.93</td>
<td>0.98</td>
<td>0.95</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

### SEM-EDX

SEM images and EDX distribution maps of Fe, Ce, and O of 5 wt.% CeO₂ sorbents calcined at 673 K (a) and 1073 K (b).

- Powders contain a mix of grains of different sizes, some are enriched in Ce, others are Ce depleted.

### MAGNETIC PROPERTIES

- **Phase diagrams** for the Fe-oxide sorbents at different temperatures.

### FORC DIAGRAMS

Map ρ(H_c, H_m) of the magnetic response of all particles in a sample with irreversible magnetizations in terms of the coercivity (switching field) H_c and magnetic interaction field H_m = H_ex distribution:

<table>
<thead>
<tr>
<th>H_c (kA/m)</th>
<th>H_m (kA/m)</th>
<th>ρ(H_c, H_m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>10</td>
<td>0.3</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>0.2</td>
</tr>
<tr>
<td>15</td>
<td>20</td>
<td>0.1</td>
</tr>
</tbody>
</table>

- The maximum close to zero (samples calcined at 673 K and 873 K of 5 and 38 wt.% CeO₂) indicates the presence of magnetite and maghemite.
- Clear contours with one central peak correspond to the dominance of the hematite (873 – 1073 K for 5 wt.% CeO₂; 573 – 1073 K for 38 wt.% CeO₂).
- Results of FORC diagrams refer to a slowing down of the transformation of iron oxides in samples with a higher content of CeO₂, and correspond well to the XRD results.
- Henkel plots obtained from virgin and hysteresis loops at RT are convex curves showing negative (diopar) magnetic interactions for all samples. The strongest interactions observed at the magnetic field H_m are close to the H_c peak of the FORC diagrams.

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