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Synthesis and Analysis Microstructural of CeO_2 Nanoparticles Using Chelating Agents

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Cerium oxide is a material with important technological applications such automotive exhaust catalysts, oxygen sensor, fuel cells among others [1]. In order to prevent the agglomeration that is commonly found by hydrothermal synthesis different chelating agents were used; citric acid, ascorbic acid and ethylenediaminetetraacetic acid (EDTA) in order to get a proper dispersion.

All the chemicals were analytical grade purity and used as received without further purification before the synthesis process. In a typical hydrothermal synthesis process, 2.0 mg of chelating agents in each particular case: citric acid ($C_6H_8O_7$), ascorbic acid ($C_6H_8O_6$) and EDTA ($C_{10}H_{16}N_2O_8$) and 2.0 g of Ce $(NO_3)_3$.$6H_2O$ were together mixed in 75 mL ethanol/ water solution (9:1), followed by a stirring for 0.5 h to form the white suspension. Subsequently it was transferred into a 100 mL Teflon/lined autoclave and heated at two different temperatures 160°C and 190°C for 24 h followed by natural cooling to room temperature. The white precipitates were washed with distilled water three times and they were dried at 80°C for 2 h. The yellow light CeO_2 was obtained after the resultant powders were calcined at 500°C for 2 h. Microstructural characterization was carried out by TEM model Hitachi 7700, SEM model JSM-7201F and X-ray diffraction.

Figure 1 shows the different morphologies and agglomerations (spherical and veils) of CeO_2 using the chelating agents. Figure 2 shows sample B1 representatives of ascorbic acid as chelating agents, the red arrows indicate spheres collapsed which can be attributed to the temperature of synthesis. XRD was also used to estimate the mean crystallite size (d_{XRD}) of the materials, using the Scherrer equation. The size of CeO_2 materials are on the nanometers scale. The types of CeO_2 obtained under different temperatures and synthesis conditions are shown in Table 1. Generally, $\varphi = d_{BET}/d_{XRD}$ is defined as a factor to reflect the agglomeration: the φ value of 1.0 indicates no agglomeration among crystallites and the φ value closer to 1 indicates a lower agglomeration, i.e. better dispersion [2,3].

References:

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[2] C Benmouhoub et al., Materials Science Forum **609** (2009), p. 189.
[3] Y-P Fu et al., Journal of the American Ceramic Society **91**(1) (2007), p. 127.

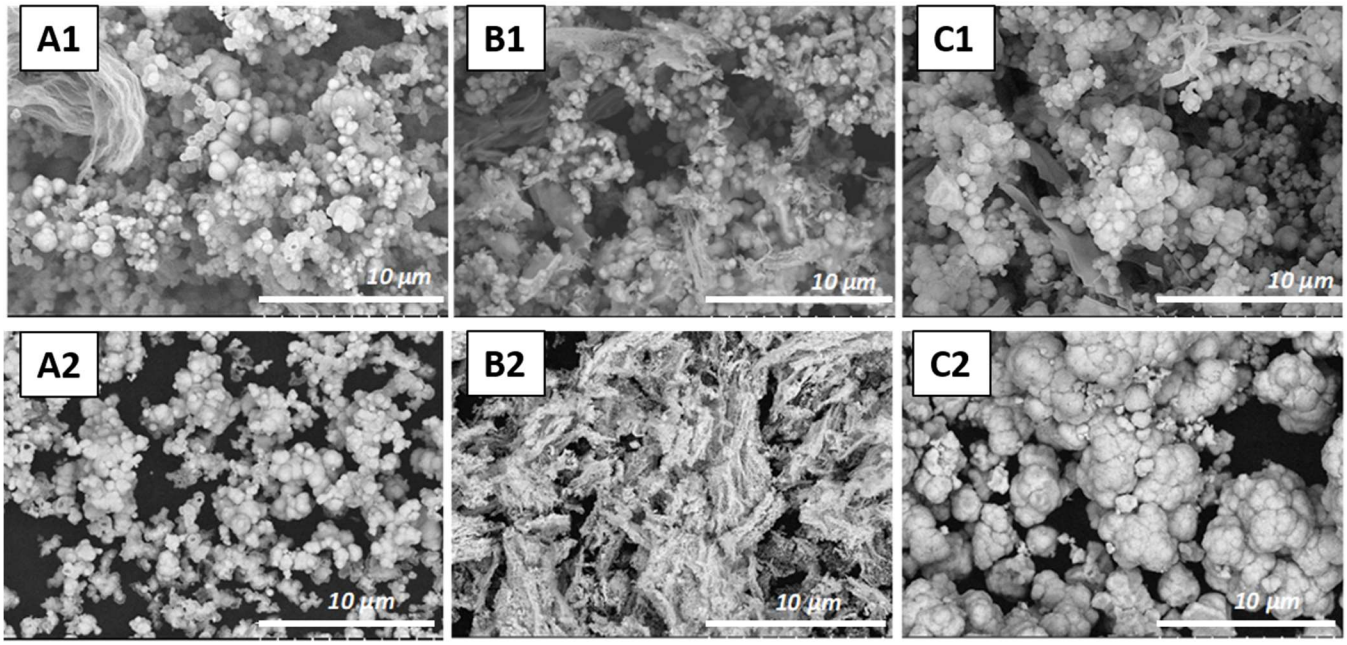

Figure 1. Micrographs SEM BSE-COM mode. Samples of CeO_2 at 160°C (A1, B1, C1) and CeO_2 at 190°C (A2, B2, C2).

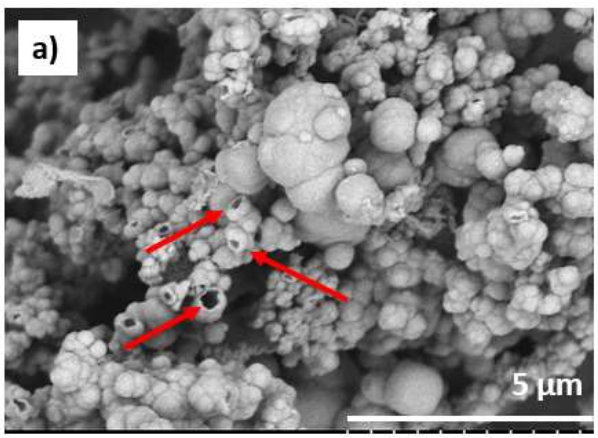

Table.1 Textural properties of CeO_2 nanoparticles

		BET results		XRD results	
Sample	pH	S_{BET} (m^2g^{-1})	d_{BET} (nm)	d_{XRD} (nm)	φ
A1	2	51.50	16.00	14	1.14
A2	2	44.59	18.48	19	0.97
B1	4	71.62	11.50	11	1.04
B2	4	65.77	12.53	15	0.83
C1	3	41.05	20.07	18	1.11
C2	3	53.13	15.51	17	0.91

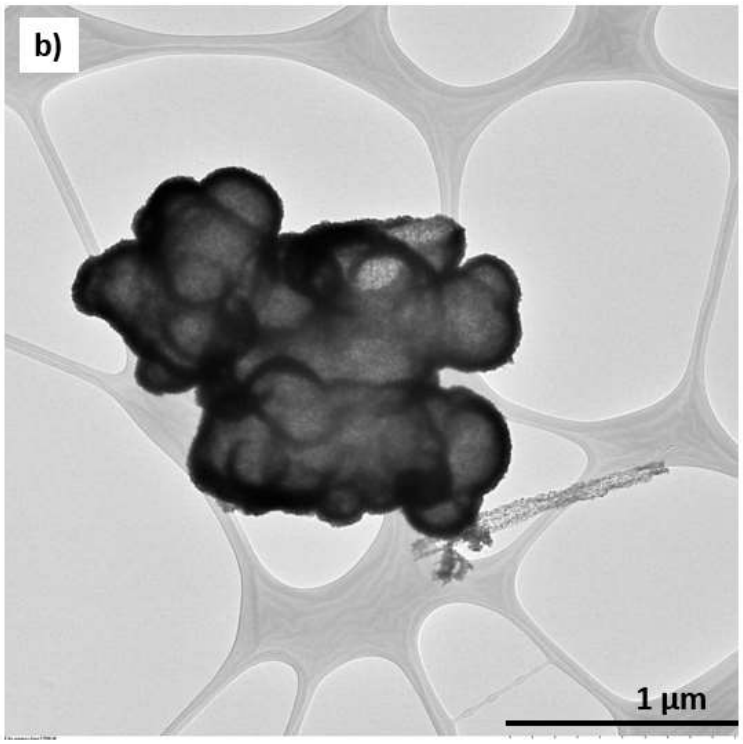

Figure 2. Micrographs: a) SEM BSE-COM mode of sample B1 shows collapsed spheres (see red arrows), b) TEM-HRTM of sample A1 shows agglomerated CeO_2 spheres.

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