doi:10.1017/S1431927619009693
Microsc. Microanal. 25 (Suppl 2), 2019

Hydrothermal Synthesis of Nanohybrid Gr-CeO_2

M. L. Camacho-Rios[1], R. A. Ochoa-Gamboa[1], W. Antunez-Flores[1], D. Lardizabal-Gutierrez[1], J. D. Cristóbal -García[1], I. Estrada-Guel[1] and R. Martínez-Sánchez[1].

[1.] Centro de Investigación en Materiales Avanzados (CIMAV), Laboratorio Nacional de Nanotecnología, Miguel de Cervantes No. 120, 31136, Chihuahua, Chih., Mexico.
* Corresponding author: luisa.camacho@cimav.edu.mx

Nanohybrids (NHs) materials are a conjugation of metal oxides with a carbonaceous coating or doping. These combinations have been pursued to enhance the original oxide performance and/or incorporate multifunctional properties. NHs have emerged as a new class of compounds that are interesting in diverse areas such as: chemistry, physics, material sciences, biology and medicine [1]. When CeO_2 and Graphite are conjugated, the resulting properties are expected to be different, for cases when one or more of the components properties will become dominant. This can be a function of the conjugation or the synthesis procedure. Such changes may be manifested in their resultant size, shape, crystalline structure, surface chemistry, etc.

Natural graphite flakes (-10 mesh, 99.9% purity from Alfa Aesar Co.) was used in this work as raw material. Graphite was processed in a high-energy SPEX 8000M mill. The balls to powder ratio was kept 5 to 1 (in weight). Milling was accomplished after a period of 8h under an inert argon atmosphere. Ceria synthesis was performed mixing 2.0 mg of chelating agent (citric acid, $C_6H_8O_7$) and 2.0 g of $Ce(NO_3)_3 \cdot 6H_2O$ in 75 mL ethanol/water solution 9:1 (in volume). The Nanohybrid Gr-CeO_2 (NH Gr-CeO_2) was prepared by mixing the above solution with the milled graphite following this route: 1.0 mg of graphite was added in the solution; the mixture was sonicated for 0.5h and heated at 160°C for 24 h. After cooling, the solid sample was filtered, washed, dried and calcined at 500°C for 2h, under a protective argon atmosphere to avoid graphite oxidation. The microstructural characterization was carried out through two electron microscopes: a Hitachi TEM model 7700 and a Jeol SEM model JSM-7201F. XRD studies were performed using a Bruker diffractometer model D8 Advance.

Fig. 1 presents two SEM images of CeO_2 and NH Gr-CeO_2, where one can notice a coral-like structure in both cases, the observed particles exhibiting a low agglomeration after graphite addition. Fig. 2 (a-b) displays SEM micrographs and EDS elemental analyses of products. There is an important concentration of carbon in the NH Gr-CeO_2 sample. Based on XRD studies, the mean crystallite size was calculated using the Scherrer equation, the determined values for CeO_2 and NH Gr-CeO_2 were 14 nm and 11 nm, respectively. In Fig. 2c we observe nanoparticles of CeO_2 with sizes lower than 20 nm, which agrees with the XRD results. As was described, the citric acid was used as a chelating agent to inhibit growth and to reduce agglomeration of the particles [2,3]. Fig. 2d shows graphite sheets decorated with CeO_2 nanoparticles on their surface. These particles were homogeneously distributed. Fig. 2e presents a STEM/EDS elemental mapping, which confirms the presence of carbon as a main component of the prepared NH Gr-CeO_2.

References:
[1] NB Saleh et al., Nanomaterials **4** (2014), p. 372.
[2] T Masui et al., Journal of Materials Science Letters **21** (2002), p. 489.
[3] Y Zang et al., Journal of Dispersion Science and Technology **28** (2007), p. 1053.

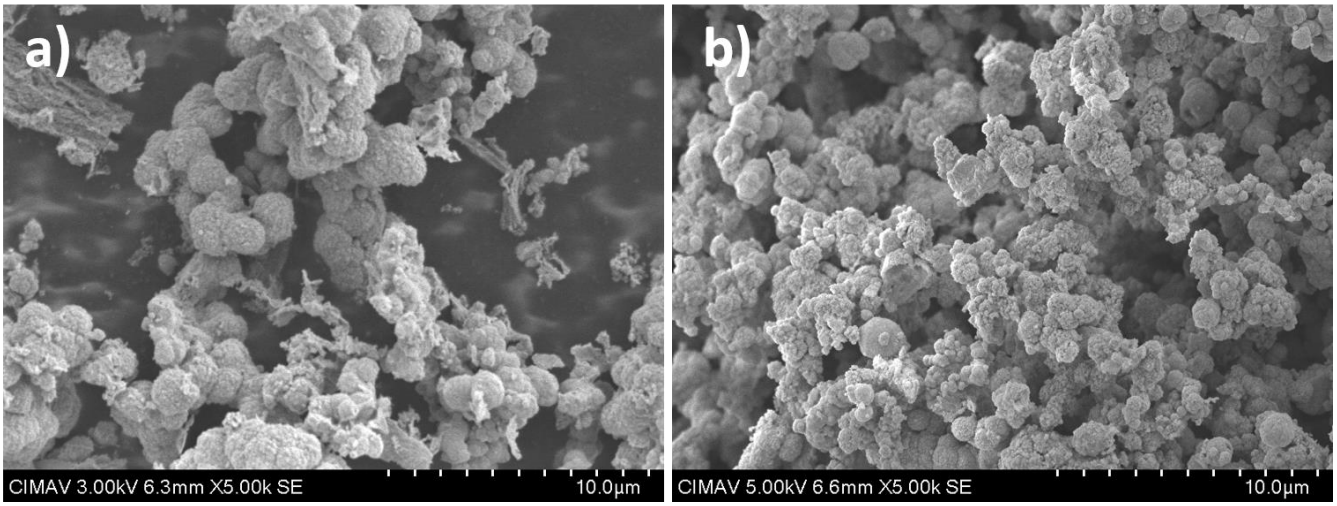

Figure 1. SEM micrographs: a) CeO_2 and b**)** NH Gr-CeO_2.

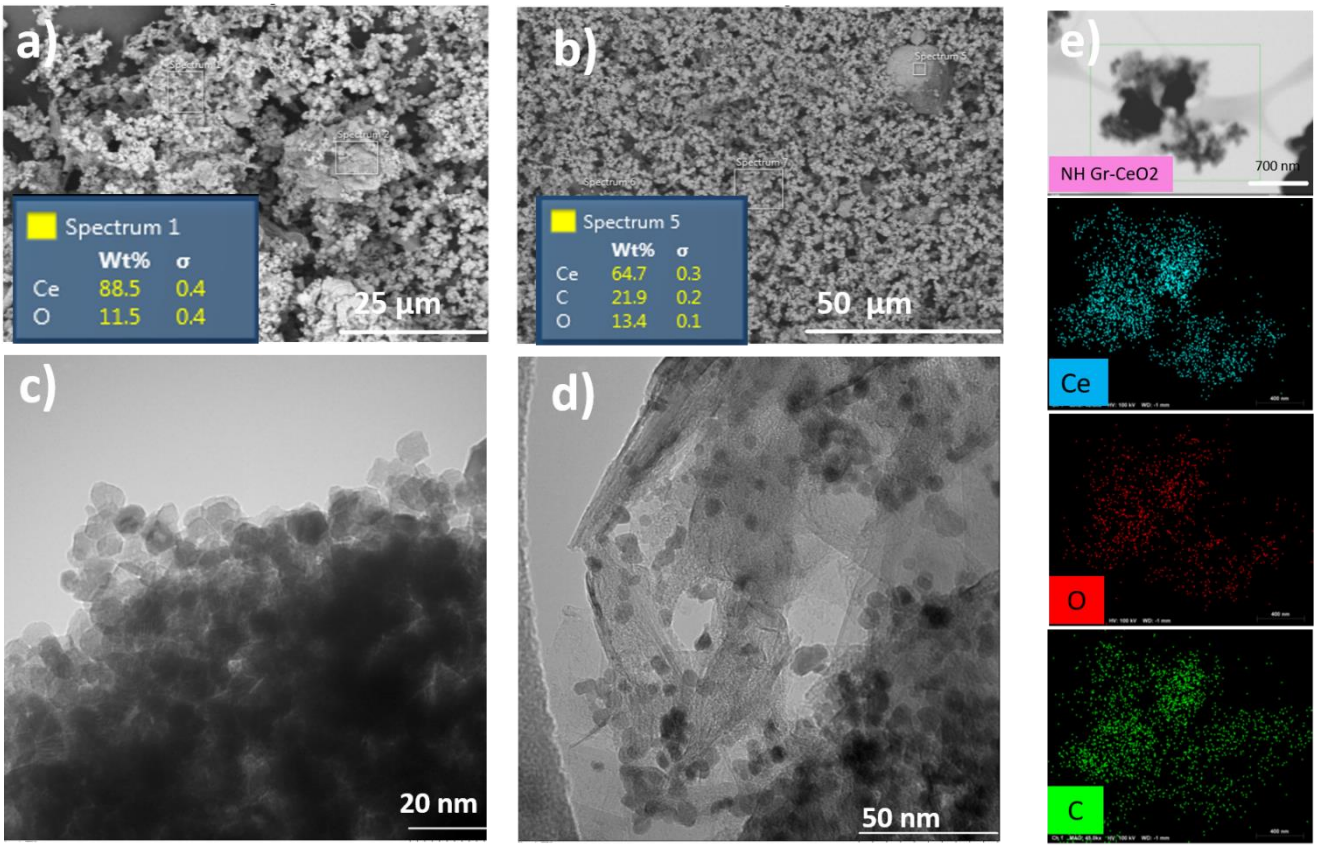

Figure 2. SEM micrographs and EDS elemental analysis: a) CeO_2, b) NH Gr-CeO_2. TEM micrographs: c) CeO_2, d) NH Gr-CeO_2, and e) TEM micrograph and corresponding EDS maps of NH Gr-CeO_2 particle.

https://doi.org/10.1017/S1431927619009693 Published online by Cambridge University Press