SYNTHESIS AND TEM CHARACTERIZATION OF LANTHANUM NANOPARTICLES BY A REVERSE MICELLAR METHOD

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Background: The synthesis of lanthanide nanoparticles is a research area of big interest, especially since these materials have very interesting optical, magnetic and catalytic properties. [1]. Lanthanide particles have been synthesized by different means but in general particles were large and oxides have been obtained. In these work we show that through a reverse micellar method we have been able to produce small Lanthanum nanoparticles, (3-5 nm) and La nanorods.

Methods: The first solution is prepared from hexadecyldimethylammonium chloride (CTAC surfactant) with benzene to form a reverse micelle solution. A second solution of La salt in an appropriate amount of water was prepared and added to the first micellar solution. Following this step a $NaBH_4$ solution was slowly added to the reaction mix, while the solution was vigorously stirred. Finally the solution is allowed to sit still, in order to separate the phases, the organic and aqueous. To the organic phase, water is added to eliminate the excess of surfactant and the phases separate again. As soon as the process was finished the grids are prepared for high resolution transmission electron microscopy (HRTEM) characterization. The samples were prepared by placing a drop of the solution on lacey formvar carbon-coated 3 mm copper grids. Electron microscopy was performed in a JEOL JEM-2010F FasTem Microscope. High resolution images were obtained at the various defocus conditions, as well as High angle annular dark field, (HAADF) images, which allowed us to clearly identify the metal particles and finally Electron Energy Loss Spectroscopy, EELS, which gave us confirmation of the chemical state of the nanoparticles.

Results: The results are shown in figures 1-3. Figure 1 shows the synthesized La^0 nanoparticles with average diameter of 3 nm; The formation of the nanophases can be appreciated in the HAADF images (Fig.2), the brightest particles corresponded to metallic Lanthanum. In this figure it is clearly shown that some of the particles are clustered together. Rods are also observed. The clustering of the particles is mainly due to the micellar structure generated during the synthesis, surface energies of the particles and the shape of the supporting film on the microscope grid. Some salts characterized as NaCl, product of the synthesis were also observed. In figure 3 a Lanthanum Nanorod obtained is shown; careful measurement of the d-spacing from the diffractograms (inset) helped us to determine that the particles were $\mathbf{La_2O_3}$.

Conclusions:

1. The Reverse micellar method was used successfully to obtain La nanoparticles in metal and oxide states of sizes between 3-5 nm.
2. Typical structures of Lanthanum in metallic state were MTPs.

3. Hexagonal La and defective particles were observed.
4. Information obtained from HAADF on larger La nanoparticles allowed us to determine that they were epitaxial on NaCl crystals.

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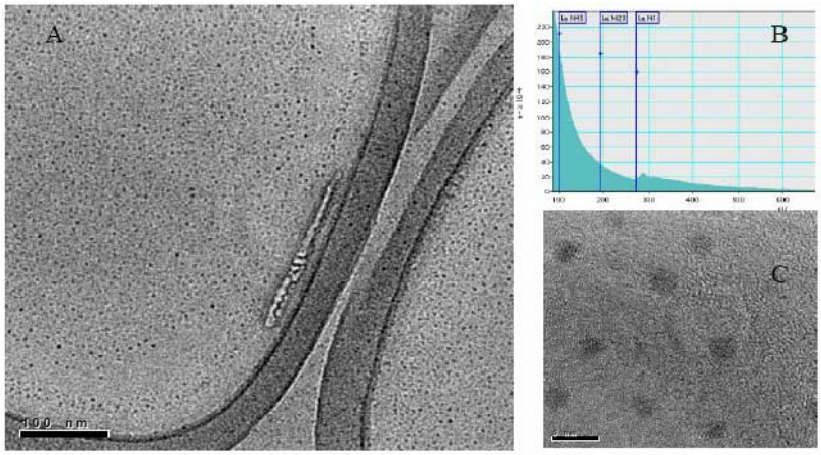

Fig. 1. A) Lanthanum (La) nanoparticles (bright Field image) B) EELS showing the characteristic La peaks; C) Higher magnification image

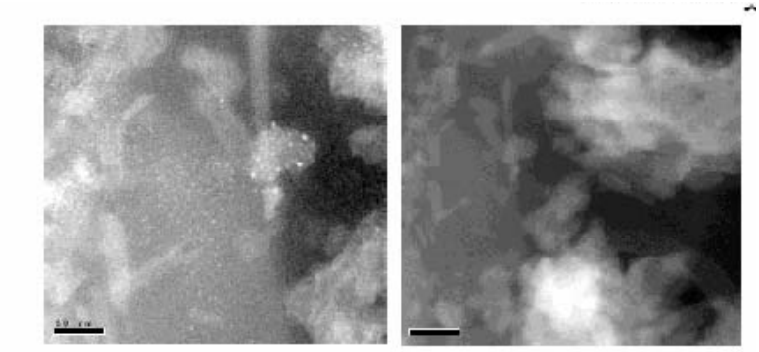

Fig.2. High-Angle Annular Dark-Field images of La sample nanoparticles, nanorods are observed.

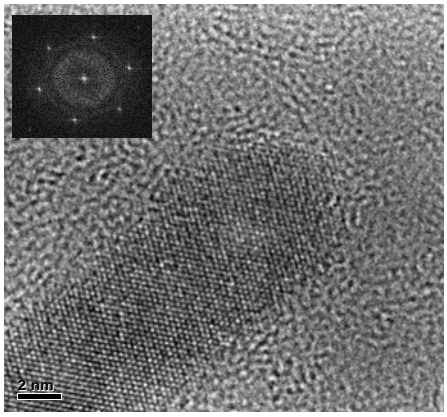

Fig.3. High Resolution Image of the tip of a Lanthanum Oxide $\mathbf{La_2O_3}$ nanorod.