

Synthesis and Characterization of La(OH)₃ Nanorods by Hydrothermal Microemulsion Method

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Abstract: La(OH)₃ nanorods with diameters of 20-40 nm and lengths of 200-300 nm were synthesized by a hydrothermal microemulsion method. The structure and morphology of the final products were characterized by X-ray powder diffraction (XRD), transmission electron microscopy (TEM), and field emission scanning electron microscope (FESEM).

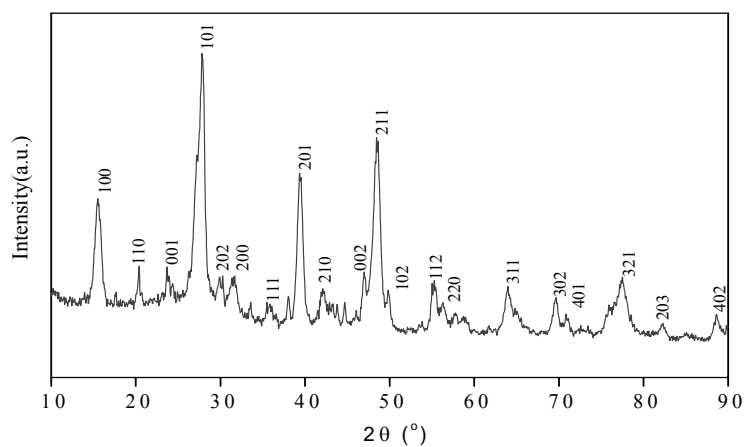
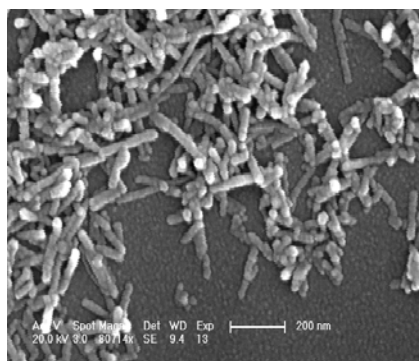
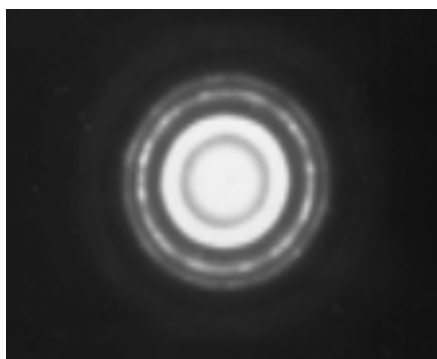
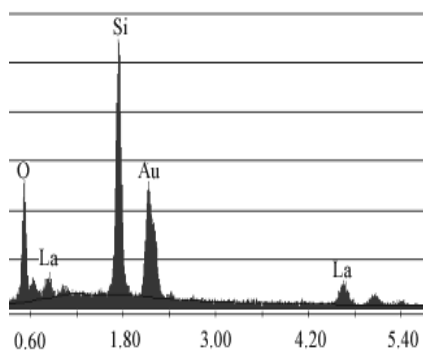
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Over the past decade, one dimensional (1D) nanostructures, such as nanotubes, nanorods, nanowires, and nanobelts have attracted considerable attention because of their special properties¹. Many methods have been developed in order to realize shape control of one dimensional nanomaterials including chemical vapor deposition (CVD), arc discharge, laser ablation, solution, catalytic assisted method, template assisted method, inverse micelles, liquid crystal *etc.*²⁻¹⁰. Of the methods used in 1D nanostructures synthesis, hydrothermal method have emerged as powerful tools for the preparation of anisotropic nanomaterials¹¹. Rare earth compounds have been used as luminescent devices, catalysts, magnets and other functional materials.

As we know, those properties with the nanometer regime might be associated with their sizes and morphologies,¹² thus if the rare earth compounds were obtained in a rod-like form, they might act as highly functionalized materials. The large ratio of surface area to volume can contribute to some of the unique properties of nanomaterials. Catalysis is one of the important applied fields of La(OH)₃, larger specific surface area and higher activity of the La(OH)₃ nanorods can improve catalytic efficiency dramatically¹³.

To the best of our knowledge, few studies have focused on the preparation of rod-like lanthanide-related compounds. Recently, Tang *et al.*¹⁴ and Deng *et al.*¹⁵ reported the synthesis of La(OH)₃ nanorods by using sol-solvothermal and hydrothermal method. Here we reported the preparation of La(OH)₃ nanorods by the hydrothermal microemulsion method.

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Figure 1 XRD pattern of $\text{La}(\text{OH})_3$ nanorod**Figure 2** TEM images of $\text{La}(\text{OH})_3$ nanorods**Figure 3** SEM images of $\text{La}(\text{OH})_3$ nanorods**Figure 4** SAED pattern of $\text{La}(\text{OH})_3$ nanorods**Figure 5** EDX image of $\text{La}(\text{OH})_3$ nanorods

The synthesis of La(OH)₃ nanorods included the following steps: 1.0 g CTAB was dissolved in 25 mL of cyclohexane, 1 mL of *n*-pentanol, and 1 mL of 0.2 mol/L La(NO₃)₃ aqueous solution, then the solution were stirred for 30 min and till to become transparent. Then 2 mL of 0.6 mol/L KOH aqueous solution was added to the above transparent solution under stirring. Afterward, the solution were stirred for another 15 min, the resulting microemulsion solution was transferred into stainless teflon-lined autoclave and heated at 160°C for 12 h. Finally, a white precipitate was collected by centrifuging, washed three times with absolute ethanol and distilled water, respectively, and dried in a oven at 60°C for 12 h.

The samples were characterized by powder X-ray diffraction (XRD) on Rigaku D/max-II B X-ray diffractometer, using CuK α radiation. XRD patterns were recorded from 10-90° with a scanning step of 0.02°. Transmission electron microscopy (TEM) images and selected area electron diffraction (SAED) patterns were taken with a JEOL JEM-2010 transmission electron microscope, with an accelerating voltage of 200 kv. The morphologies of the sample were characterized by a Micrio FEI PHILIPS XL 30 ESEM FEG scanning electron microscope (ESEM), and energy dispersive X-ray (EDX) spectrum of the La(OH)₃ nanorods sample was obtained also using the same instrument. **Figure 1** show powder XRD pattern of the as-synthesized La(OH)₃ nanorods. All of the reflections of the XRD patterns of the as-synthesized La(OH)₃ nanorods can be easily indexed to a pure hexagonal crystalline La(OH)₃(JCPDS41-4019), indicating the formation of La(OH)₃. La(OH)₃ lattice constants calculated from the XRD data are $a=6.526 \text{ \AA}$ and $c=3.860 \text{ \AA}$, consistent with those of bulk La(OH)₃ (JCPDS 41-4019). The morphologies and size of the La(OH)₃ products were investigated by TEM and field emission scanning electron microscope (FESEM). The results are shown in **Figure 2** and **Figure 3**. It can be seen that La(OH)₃ products are almost entirely nanorods with diameters of 20-40 nm, and lengths of 200-300 nm. **Figure 4** is the corresponding SAED pattern, taken on the La(OH)₃ nanorods. It is observed that the SAED pattern consists of many diffraction circles, which shows La(OH)₃ nanorods is polycrystal. Energy-dispersive X-ray (EDX) spectroscopy analysis (**Figure 5**) showed the sample contains La and O; but no K was detected, indicating the final product was free of elemental K and in high purity (the peaks corresponding to the Si atom and Au atom, respectively, originate from the silicon plate and spraying gold).

In summary, we have developed a simple and novel route to prepare La(OH)₃ nanorods by hydrothermal microemulsion method. This simple approach may provide a novel route for synthesis of this nanostructured material in a controlled manner for many important applications in nanotechnology.

References

1. J. Hu, M. Ounyang, C. M. Lieber, *Nature*, **1999**,399, 48.
2. C. C. Chen, C. C. Yeh, *Adv. Mater.*, **2000**,12 , 738.
3. Z. G. Bai, D. P. Yu, H. Z. Zhang, *et al.*, *Chem. Phys. Lett.*, **1999**,303, 311.
4. Y. Wu, P. Yang, *Chem. Mater.*, **2000**, 12, 605.
5. M. Yazawa, M. Koguchi, A. Muto, M. Ozawa, K. Hiruma, *Appl.Phys. Lett.*, **1992**, 61 , 2051.
6. Y. C. Choi, W. S. Kim, Y. S. Park, *et al.*, *Adv. Mater.*, **2000**, 12, 746.

7. (a) X. F. Duan, C. M. Lieber, *Adv. Mater.*, **2000**, 279, 208.
(b) A. M. Morales, C. M. Lieber, *Science*, **1998**, 279, 208.
8. T. J. Trentler, K. M. Hickman, S. C. Goel, *et al.*, *Science*, **1995**, 270, 1791.
9. J. D. Holmes, K. P. Johnston, R. C. Doty, B. A. Korgel, *Science*, **2000**, 287, 1471.
10. (a) M. H. Huang, A. Choudrey, P. Yang, *Chem. Commun.*, **2000**, 12, 1603.
(b) J. Zhu, S. Fan, *J. Mater. Res.*, **1999**, 14, 1175.
11. G. R. Patzke, F. Krumeich, R. Nesper, *Angew. Chem. Int. Ed.*, **2002**, 41, 2446.
12. S. Nie, S. R. Emory, *Science*, **1997**, 275, 1102.
13. A. Tsubouchi, T. C. Bruice, *J. Am. Chem. Soc.*, **1995**, 117, 7399.
14. B. Tang, J. C. Ge, C. J. Wu, *et al.*, *Nanotechnology*, **2004**, 15, 1273.
15. Y. Deng, J. B. Wu, J. Liu *et al.*, *Journal of Physics and Chemistry of Solids*, **2003**, 64, 607.
16. M. H. Cao, Y. H. Wang, C. X. Guo, Y. J. Qi, C. W. Hu, *Langmuir*, **2004**, 20, 4784.

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