

Low Temperature Materials Synthesis Based in the Benzenetricarboxylate Method

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Rare earth (RE) benzenetricarboxylate (BTC) complexes (1,3,5-benzenetricarboxylic acid - TMA and 1,2,4-benzenetricarboxylic acid - TLA) present favorable thermal properties to prepare oxides at low temperatures [1]. The luminescent nanophosphors were obtained by annealing the complexes from 500 to 1000 °C. The thermogravimetric curves of the [RE(BTC):Eu³⁺ (x %)] complexes (RE³⁺: Y, Gd and Lu; x: 0.1, 0.5, 1.0 and 5.0) show that the organic moiety of the complexes decomposes in a single-step from 430 to 580 °C, allowing the formation of RE₂O₃ after 1 h annealing at 500 °C. The decomposition temperature decreases for all complexes with increasing Eu³⁺ concentration.

The X-ray powder diffraction patterns confirm the formation of the cubic RE₂O₃ phase, indicating the total decomposition of the organic phase during annealing. The crystallinity of the oxides increases with the annealing temperature. According to the Scherrer's equation, the crystallite sizes of Y₂O₃:Eu³⁺ (TMA precursors) grow from 6 to 52 nm between 500 and 1000 °C for Y₂O₃:Eu³⁺.

The luminescent properties were investigated by the excitation and emission spectra as well as lifetime measurements of the ⁵D emitting level of Eu³⁺. The number of lines for the ⁵D→⁷F₁ transition indicates the presence of Eu³⁺ in two sites (C₂ and S₆). An intense hypersensitive ⁵D→⁷F₂ transition due to the non-centrosymmetric C₂ site was observed. Finally, [RE(BTC)] complexes emerge as suitable precursors for the preparation of Eu³⁺-doped RE₂O nanophosphors at low temperature, yielding strong luminescence with quantum efficiency up to 96 %.

[1] E.R. Souza, I.G.N. Silva, E.E.S. Teotonio, M.C.F.C. Felinto, H.F. Brito, J. Lumin. 130 (2010) 283-291.