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Synthesis of $CaTiO_3$ nanoparticles using Hydrothermal Microwave Method

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Abstract – There were obtained nanoparticles of calcium titanates by using a simple microwave-hydrothermal method (HTMW). The obtained powder was characterized by X-ray diffraction (XRD), spectroscopy Raman and photo-luminescence (PL). All the peaks at the XRD pattern (Fig. 1) can be indexed to a pure orthorhombic phase of $CaTiO_3$ (81-0562), which is confirmed by the Raman data. The PL spectrum (Fig. 2) is typical of a multiphonon process and the maximum emission is centered at 530 nm (green).

The hydrothermal microwave (HTMW) method is an alternative synthesis process developed recently to prepare nanoparticles. It is a low-temperature and high reacting rates method which permit process powdered ceramic materials in short times with uniform microstructure. Due to the short time and temperature reactions this technique allow to control unwanted grain growth and the final particle-size [1,2].

The $CaTiO_3$ (CT) nanoparticles were synthesized using a solution (0.01 mol) of calcium chloride ($CaCl_2$.2H2O) in deionized water under constant stirring and nitrogen flowing out to avoid carbonate formation. Titanium (IV) isopropoxide ($C_{12}H_{28}O_4Ti$), 3.1 ml, and KOH (50 ml) were adjoined to the solution. The reaction mixture was placed into a Teflon autoclave which was sealed and placed in the HTMW system (Fig. 3), a modified domestic microwave (2.45 GHz and maximum power of 800 W). The solution was heated to 140°C (at 140°C/min) and was maintained at this temperature for 40 min under a pressure between 3 and 4 atm.

The X-ray diffraction pattern (Fig. 1) of the crystalline product was identified using the PDF file (81-0562) as being a pure orthorhombic calcium titanate ($CaTiO_3$). The Raman spectrum confirms this result. The photoluminescence profile (Fig. 2) is typical of a multiphonon process, i.e., the emission that occurs by several paths, involving numerous states within the band gap of the material [1]. Due to the missing of organic material in the synthesis process, the PL emission is associated to the material structure defects [3]. The PL spectrum indicates an energy band in the visible region around the green light range, with the maximum emission centered at 530 nm. The mean crystallite size of $CaTiO_3$ calculated using Scherrer equation is 40 nm [4].

These results show that nanoparticles of pure calcium titanate can be synthesized at low temperature in a short reaction time by using the HTMW method. The material has orthorhombic structure, mean cristaline size of 40 nm and present photoluminescence emission.

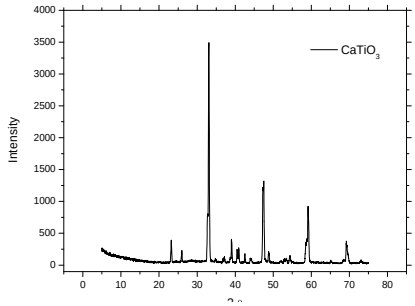

Figure 1: X-ray diffraction.

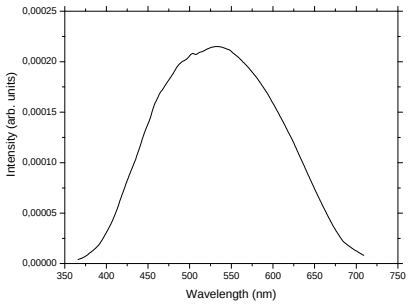

Figure 2: Photoluminescence spectrum.

Figure 3: Modified domestic microwave.

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