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Synthesis of $Ba_xCa_{1-x}TiO_3$ Nanoparticles using Microwave-assisted Hydrothermal Method

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Abstract – Microwave-assisted hydrothermal (MAH) method was used in the synthesis of $Ba_xCa_{1-x}TiO_3$ (0, 25, 50, 75 e 100%) nanoparticles. $C_{12}H_{28}O_4Ti$, $BaCl_2.2H_2O$, $CaCl_2.2H_2O$ and KOH were used as precursors. The solution was heated inside of closed vessel in a microwave oven at 140ºC, with rate of 140ºC/min for 40 minutes. The ceramic powder was characterized by X-ray diffraction (XRD), Raman Spectroscopy (RS) and Photoluminescence (PL). Although the XRD pattern does not show the tetragonal structure the Raman spectrum shows characteristic band of this symmetry. The photoluminescence spectrum presents emission band between 400 and 550nm.

The barium titanate ($BaTiO_3$) perovskite, a ferrelectric material, has been extensively studied due to its several possible applications such as electronic and optical devices. The piezoelectricity properties of $BaTiO_3$ perovskite is improved with the Ba by Ca substitution. As a consequence of this, the $Ba_xCa_{1-x}TiO_3$ solid solution has attracted great attention for its use in laser systems, electro-optic materials for various applications [1]. The $Ba_xCa_{1-x}TiO_3$ (BCT) nanoparticles were synthesized using a solution of barium chloride ($BaCl_2$.2H2O) and calcium chloride ($CaCl_2.2H_2O$ in deionized water under constant stirring. 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 operating with 2.45 GHz and maximum power of 800 W. The MAHmethod 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 allows to control unwanted grain growth and the final particle-size [2]. 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. **Figure 1** illustrates the XRD patterns of all compositions in which it can be identified $CaTiO_3$ (82-0229), $BaTiO_3$ (83-1878) and solid solutions between them. Also were identified Ca and Ba carbonates. In the composition x = 25% only Ca carbonate was formed. Although in the phases rich in Ba the XRD patterns can be identified as tetragonal or cubic structure the RS spectrum **(Fig. 2)** confirms the tetragonal symmetry. The bands near 713, 519, 305, 270 and 180 are characteristics of tetragonal $BaTiO_3$ [2] and decrease with the Ca concentration increasing. The band around of 1060 cm^{-1} is attributed to carbonates formation and agrees with the XRD data [1]. An intense and broad PL band **(Fig. 3)** report a maximum centered around 450 to 550 nm by a multiphotonic process for BCT samples. These results show that nanoparticles of pure $Ba_xCa_{1-x}TiO_3$ can be synthesized at low temperature in a short reaction time by using the MAH method.

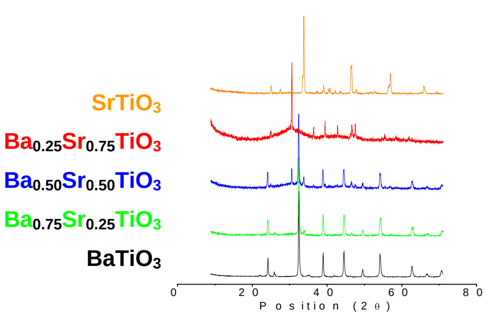

Figure 1: X-ray diffraction.

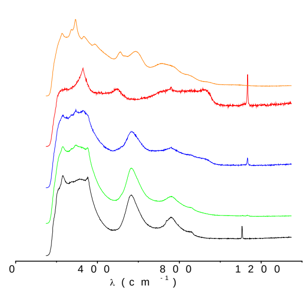

Figure 2: Raman spectrum.

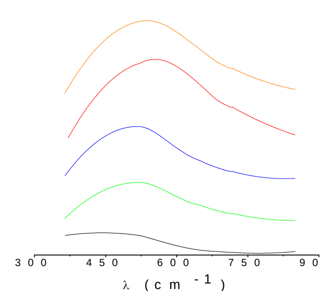

Figure 3: Photoluminescence Spectrum.

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