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$Ba_2In_2O_5$ Solid Electrolytes: pH controlled chemical Synthesis and Characterization

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Abstract – $Ba_2In_2O_5$ solid electrolytes were synthesized by polymeric precursor method at different pH conditions. Low temperature thermally treated materials were studied by Thermogravimetric Analysis and Fourier Transform Infrared Spectroscopy (FTIR). The main results showed a different thermal decomposition process for the material obtained at pH=6. Carbonyl absorption bands are shifted to lower frequencies, when compared to the same bands of the free citric acid, indicating a dependence of preparation parameters on the coordination of the citrate with the metal ions.

Oxygen ion conductors have been extensively studied due to their potential technological application, such as oxygen sensors, electrolytes in solid oxide fuel cells, and oxygen permeable membranes. Oxide ceramics with cubic perovskite or related structures may exhibit high oxygen-ion conductivity. The mixed oxide $Ba_2In_2O_5$ presents an orthorhombic structure from room temperature up to 925 °C with cell parameters a=0.608 nm, b=1.679 nm and c=0.589 nm. Above this temperature, an order–disorder phase transition occurs and the crystalline structure changes to tetragonal symmetry. A second-order phase transition at 1040 °C turns the crystalline structure to cubic perovskite, when the anionic vacancies are in a fully disordered state, and the mixed oxide shows pure ionic conduction with unitary ionic transport number[1]. Great attention has been given over the last few years to the effects imparted by the physical properties of the particles, for example, the influence of diameter, shape and size distribution on bulk ceramics and thin films properties[1]. The sol- gel procedure was reported as an important way to control de size distribution on bulk ceramics, associated to the metal/ citric acid ratio[2]. The main purpose of this work is control the particle size of mixed oxide $Ba_2In_2O_5$ with orthorhombic structure by a refined pH well-controlled chemical synthesis.

The materials were obtained by polymeric precursor method at different pH (6, 7, 8, 9), with further thermal treatment at 200°C. After that, the samples were studied by thermogravimetric analysis, Fourier Transform Infrared Spectroscopy and X-ray powder diffraction. Figure 1 shows DTA curve for all samples and the material obtained with pH=6 shows three exothermic peaks around 462°C, while the samples obtained in higher pH conditions shows only one exothermic peak at the same temperature. FTIR spectra for the resulting samples (Figure 2) present an asymmetric stretching vibration *u* as (COO-) in 1650 and 1550cm^{-1} range, while a symmetric stretching vibration *u* as (COO-) occurs as an intense sharp band at 1385 cm^{-1}. By analysis of the FTIR spectra vs. pH synthesis conditions, it is observed that all of the carbonyl absorption bands are shifted to lower frequencies, when compared to those for the free citric acid, demonstrating the pH dependence of the coordination of the citrate with metal ions. Such effect could provide material preparation with diminished particle size as a result of the coordination of the citrate with metal ions.

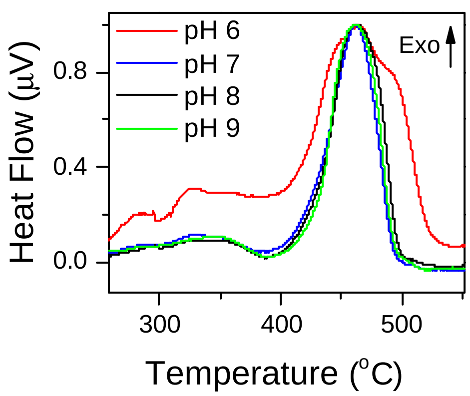

Figure 1: DTA thermograms for $Ba_2In_2O_5$ prepared by polymeric precursor method at different pH synthesis condition.

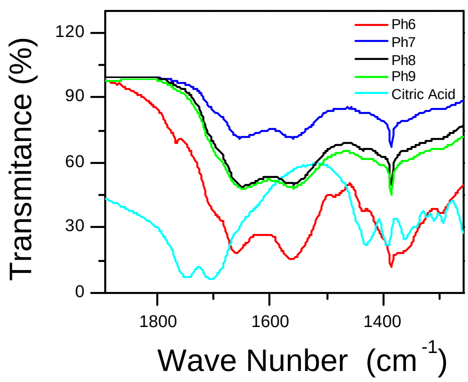

Figure 2: FTIR spectra for $Ba_2In_2O_5$ prepared by polymeric precursor method at different pH synthesis condition.

References

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[2] E. R. Leite, I. T. Weber, E. Longo and J. A. Varela, Advanced Materials 12 (2000) 965 -968