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Synthesis of lithium cobaltite ($LiCoO_2$) prepared by solid state and sol-gel acryl amide polymerization reaction.

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Sol-gel acrylamide and solid state reactions were used to prepare a lithium cobaltite. The influence of different synthesis and the combustion process have influence in morphological properties and grain size of final product. Thermal analysis of gel and mixture of metal oxides show clear differences in its formation temperature. Scanning electron microscopy (SEM) images of calcined gel show the formation of nanoscopic crystals.

The preparation of ceramics by the sol-gel process dates from 1950; at that time Della and Rustum Roy [1] prepared a number of compositions from silicon tetraethoxide and metal nitrate salts. This method gave products more homogeneous than other obtained after successive melting and crushing operations of samples from individual oxides. When powders are produced by the sol-gel method, the temperature of synthesis of complex compounds decreases by 150 – 200°C [2] (fig. 1) and the particles in this case have nanosizes (fig. 2).

To prepare the gels we used the following process [3]. Solutions with each metal Li and Co was prepared separately, following by adding of EDTA solution. Both solutions were mixed and finally the acrylamide monomer (99%) was added sequentially with the cross-linker N,N0-methylenebisacrylamide(99%) and a thermo-chemical initiator α-α- azoisobutyronitrile (AIBN, 98%) under heating and vigorous stirring. With this processes a white gel was obtained. The gel was dried on microwave oven, and then calcined at 550 ◦C for 1 h.

Figure 1, shows the thermogravimetric TGA and the differential scanning calorimetry DSC analysis for both techniques of synthesis. In DSC and TGA curves for the solid state reaction (fig. 1a), high temperature (around 700°C) was observed to formation of $LiCoO_2$, also evidence the formation of other phases or polymorphs. While the thermal analysis performed on gel shows low temperatures around 500 ° C and there are not formation of secondary products. Red line in fig.2b show a peak that correspond to transition from amorphous solid gel to crystalline solid, this is an exothermic process, and results in a peak in the DSC signal.

Figure 2 shows SEM images of the dried gel in a microwave oven. It was observed small-sized crystals of the order of micrometers. This indicates that during the hard drying stage there was formation of some crystals. The pattern of X-ray powder diffraction (XRD) in the figure 2b corresponds to the material obtained after a slow heat for 3 days.

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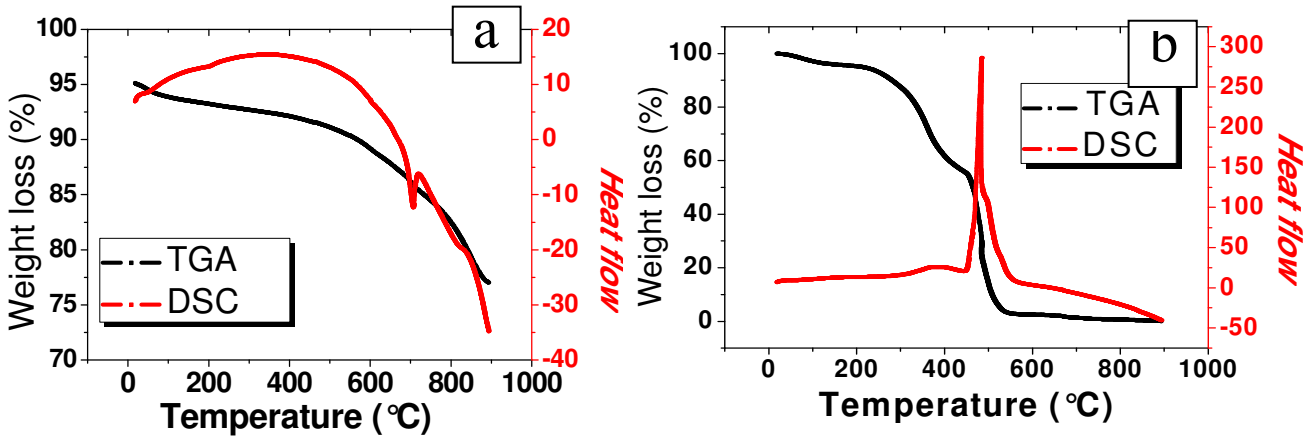

Fig.1. a) TGA and DCS analysis of $LiCO_3$ and CoO mixture. b) TGA and DSC analysis of dry polyacrylamide gel containing Li + and Co2+ cations

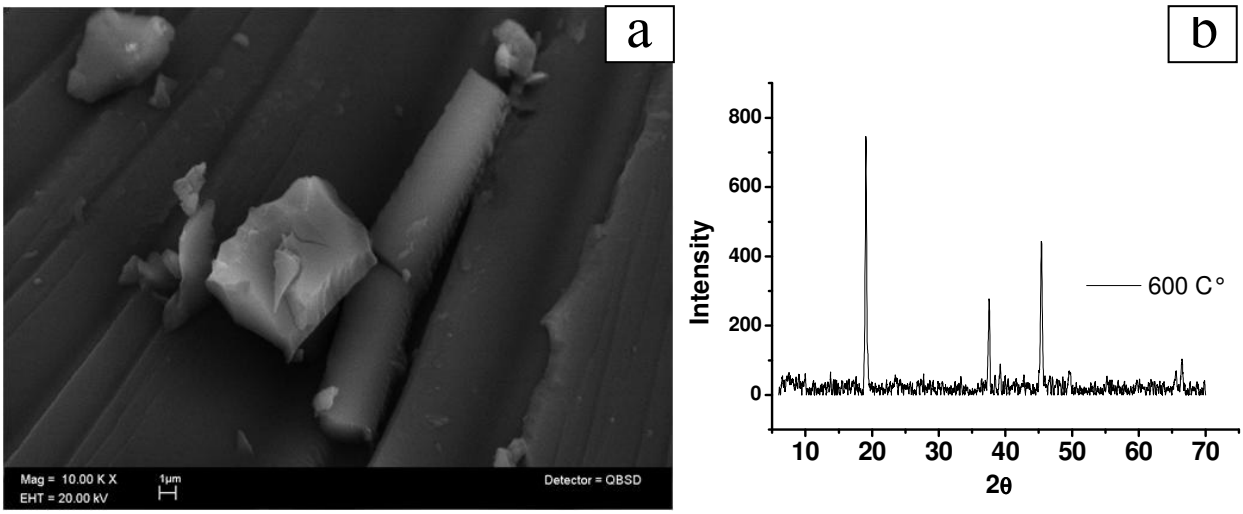

Fig.2. a) SEM images of small crystal in dry gel. The magnification used to get this image was 10.00 Kx b) XRD pattern of complex LiCoO2 that was obtained after slow heating.

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