In the last years, extensive research has been devoted to develop novel materials and structures with high electrochemical performance for intermediate-temperatures solid-oxide fuel cells (IT-SOFCs) electrodes. In recent works, we have investigated the structural and electrochemical properties of nanostructured La₀.₆Sr₀.₄CoO₃ (LSCO) and La₀.₆Sr₀.₄(Co;Fe)O₃ (LSCFO) cathodes, finding that they exhibit excellent electrocatalytic properties for the oxygen reduction reaction [1,2]. These materials were prepared by a pore-wetting technique using polycarbonate porous membranes as templates. Two average pore sizes were used: 200 nm and 800 nm. Our scanning electronic microscopy (SEM) study showed that the lower pore size yielded nanorods, while nanotubes were obtained with the bigger pore size. All the samples were calcined at 1000°C in order to produce materials with the desired perovskite-type crystal structure. In this work, we analyze the oxidation states of Co and Fe and the local atomic order of LSCO and LSCFO nanotubes and nanowires for various compositions by X-ray absorption spectroscopies. For this purpose we performed XANES and EXAFS studies on both Co and Fe K edges. These measurements were carried out at the D08B-XAFS2 beamline of the Brazilian Synchrotron Light Laboratory (LNLS). XANES spectroscopy showed that Co and Fe only change slightly their oxidation state upon Fe addition. Surprisingly, XANES results indicated that the content of oxygen vacancies is low, even though it is well-known that these materials are mixed ionic-electronic conductors. EXAFS results were consistent with those expected according to the rhombohedral crystal structure determined in previous X-ray powder diffraction investigations.


**Keywords:** XAFS, IT-SOFC cathodes, Perovskites