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Synthesis and Characterization of Porous Carbon Materials Modified with $NIFe_2O_4$ for Applications in Lithium-Ion Batteries

Raviolo Sofía[1], Amiune Nicolás[2], Carraro María Paola[3], Luque Guillermina Leticia[4], Eimer Griselda Alejandra[3], Sapag Karim[5] and Oliva Marcos Iván[6*]

[1.] Instituto de Física Enrique Gaviola, Córdoba Argentina.
[2.] FaMAF, Universidad Nacional de Córdoba, Córdoba Argentina.
[3.] Centro de Investigación y Tecnología Química (Citeq) - UTN FRC – Conicet, Córdoba Argentina.
[4.] Instituto de Investigaciones en Físico-Química de Córdoba (INFIQC) - UNC – Conicet, Córdoba Argentina.
[5.] LabSop Conicet – UNSL, San Luis Argentina
[6.] FAMAF, Universidad Nacional de Córdoba - IFEG, CONICET, Córdoba Argentina.
*Marcos Oliva: omarcos@famaf.unc.edu.ar

The present energetic situation is in a transition process that involves leaving fossil fuel as an energy resource to employ a more friendly environmentally one. To that end, it is necessary to improve the current methods of energy-storing and working in their development. Li-Ion batteries are one of the most promising ways of energy storage. In this work, we focus to improve the performance of such systems by modifying the active material in the anode. In particular, we researched about ordered mesoporous structured carbons functionalized with $NiFe_2O_4$.

With the aforementioned objective, it was started by synthesizing employing the sol-gel method [1], a matrix of ordered pores of silicon oxide (SBA-15). From which, after successive impregnations with sucrose and calcination in an inert atmosphere, a porous carbon matrix CMK-3 was obtained. These highly ordered two-dimensional porous materials have a uniform pore diameter, large surface area and remarkable thermal and electrical conductivity [2, 3]. The use of these materials as anodes in lithium-ion batteries led to a significant increase in the conduction of both lithium ions and electrons [4].
The carbon materials were modified with a $NiFe_2O_4$ spinel, using the wet impregnation method. The use of this spinel is grounded on the fact that it has a high theoretical capacity (915mAh/g) and that both Ni and Fe are low-cost and abundant elements (which is important for the possible applicability to the industry) [5]. The resulting materials were characterized by the following techniques: Low and high angle x-ray diffraction (DRX), scanning and transmission electron microscopy (SEM and TEM), elemental analysis (EDX), nitrogen adsorption isothermal analysis with BET model, ultraviolet-Visible diffuse reflectance spectroscopy (UV-vis), programmed temperature reduction (TPR), X-Ray emitted photoelectron spectroscopy (XPS) and galvanostatic cycling of electrochemical cells.

The synthesized hexagonal mesoporous structure of SBA-15 silicate can be observed directly in Figure 1, which is one of the images obtained by TEM. From this micrograph was determined the lattice parameter $a_0 = (11.8 \pm 0.2)$ nm. An SEM image of the CMK-3 impregnated with $NiFe_2O_4$ (in a proportion of 23% w/w) is shown in Figure 2. In which can be saw that the surface of the CMK-3 is coated by NiFe2O4 homogeneously, which means that, there is a good contact area between both materials facilitating the arrival of ions and electrons to the latter. For the evaluation of the electrochemical behavior of these materials acting as anodes of lithium-ion batteries, electrochemical cells with the synthesized materials were prepared. First, the following were used as anode: CMK-3, CMK-3 modified with $NiFe_2O_4$ 23% w/w (CMK/NFO (23)) and CMK-3 modified with $NiFe_2O_4$ 50% w/w (CMK/NFO (50)). An increase in gravimetric capacity was observed as the percentage of $NiFe_2O_4$ increased, but also a significant decay could be detected with cycling. For this reason, another cell was

prepared with CMK/NFO (50), but in this case using CMC as a binder instead of conventional PVDF, which resulted in a marked improvement in the cyclability of the cell. In the case of the PVDF cell, a capacity of 300mAh/g was obtained after 100 cycles, while in the CMC cell 400mAh/g was obtained, which is greater than the theoretical capacity of graphite (which is one of the most commonly used materials as anode). This improvement may be associated with the CMC binder relieving the volumetric changes that $NiFe_2O_4$ undergoes and thus maintaining the integrity of the electrode. Besides, a final electrode consisting of a physical mixture of CMK-3 and $NiFe_2O_4$ nanoparticles, synthesized separately, was prepared by comparison in 50% w/w ratio with a CMC binder. This cell showed a notable decay in the capacity with the number of cycles, obtaining the same as the CMK-3 after 100 cycles. This behavior could be justified to a loss of contact between the CMK-3 and the $NiFe_2O_4$ particles during successive charges and discharges.

The modifications of the carbonaceous material allowed us to obtain an increase in the capacity and an improvement in the cyclability of the cells. Finally, the CMK/NFO(50) synthesized by wet impregnation, using the CMC binder, was the one that presented the best electrochemical performance compared to the other electrodes.

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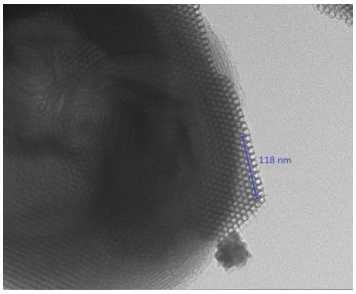

Figure 1: TEM image of the SBA-15 and segment used in the determination of the lattice parameter

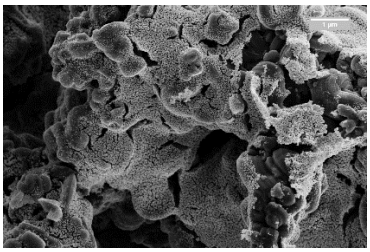

Figure 2: SEM image of CMK-3 impregnated with 23% (p/p) of $NiFe_2O_4$.

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