

Insights into Novel Solid Materials, their Recyclability and Integration

into Li Polymer Batteries for EVs. Future research directions in this field.



S O M A B A T - Advanced workshop,

Timisoara, July 4-5, 2012



## **SYNTHESIS AND CHARACTERIZATION OF POROUS CARBON XEROGELS AND ORDERED MESOPOROUS CARBONS FOR ANODE MATERIALS IN LI-BASED BATTERIES**

**Alexandre F. LEONARD, Jean-Paul PIRARD, Nathalie JOB**

*Université de Liège, Laboratoire de Génie Chimique (B6a), B-4000 Liège, Belgium  
alexandre.leonard@ulg.ac.be*

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### **Abstract**

In the aim of developing new high performance C-based anode materials for Li-ion batteries, carbon xerogels (hard carbons) as well as ordered mesoporous carbons (OMC) are promising candidates since their specific capacities widely exceed that of conventional graphitic structures. Nevertheless, hard carbons are also well-known to exhibit quite high irreversible capacity losses due to their intrinsic high microporosity[1]. In order to reduce such losses and to enhance the cycling performances, the structural and textural characteristics of such materials need to be carefully controlled. OMC in turn still remain difficult to prepare under reasonable time constraints and in green conditions, say aqueous syntheses for instance.

The polymerization, drying and pyrolysis of aqueous Resorcinol-Formaldehyde gels lead to carbon materials with a dual porosity, i.e. they are composed of microporous nodules delimiting meso-or macroporous voids, the average size of which depends on both the composition of the precursor solution (pH, mainly) and the drying procedure.[2] The application of such xerogels as anode component is however not straightforward since too a high microporosity can induce considerable irreversible capacity losses and too small mesopores hinder the proper chemical diffusion of lithium ions within a bulk electrode material. The latter is often a rate-limiting step and optimized transport pathways could be provided by creating large mesopores or even macropores within the microporous carbon structure.[3-4]

Here we report on the control of the textural characteristics of meso-microporous RF xerogels prepared by vacuum drying procedure. The improvement of accessibility in the framework was achieved (i) by adjustment of the pH of the RF precursor solution and (ii) by addition of a block-copolymer non-ionic surfactant (Pluronic F127) to the reaction mixture prepared with different Resorcinol/Na-Carbonate (R/C) molar ratios. The

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micropore volume was tuned upon addition of a supplementary aqueous carbon precursor, either via direct synthesis or by post-modification of (i) dried and (ii) pyrolysed xerogels.

Investigation of mesopore size adjustment shows that the structural features of the final pyrolyzed carbons remains unchanged, whereas the mesopore sizes can be enlarged by decreasing the pH of the precursor solution. Indeed, the lower pH leads to larger primary carbon nodules, delimiting larger voids in-between them. A more fine-tuning of mesopore sizes can be achieved upon addition of the non-ionic block-copolymer surfactant. In this case, it is believed that the surfactant acts as a scaffold preventing the structure from shrinking during the drying procedure. The increase in mesopore sizes is accompanied by an enlargement of the volume of pores  $>7.5$  nm, determined by Hg intrusion porosimetry, demonstrating an increased accessibility within the pore framework, with values that become near those found for cryogels and aerogels. Moreover, all of these features are maintained when the synthesis is scaled-up to 15 g of final porous carbon material.

The micropore volume of these materials can be decreased and tuned upon impregnation with a secondary carbon precursor. It is shown that micropores can selectively be filled at least partially, but also that the particle sizes of the starting xerogels govern the success of this route. Moreover, the final pyrolysis step inevitably induces the development of micropores.

Ordered Mesoporous Carbons offer a complementary strategy towards the development of high-performance anodes for Li-based batteries [4-5]. Indeed, such materials have been shown to offer high capacities and long-lasting cycling performances. The last part of the talk will highlight the recent developments made in the field of OMC synthesis via rapid aqueous routes.

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