

### Nanostructured carbon materials for energy storage applications

**Abstract** Nanostructured carbon and carbon hybrid materials are of great interest in many applications such as energy storage, adsorption and catalysis. The development of eco-friendly, simple and cost-effective processes allowing to control the carbon characteristics are important challenges to achieve in order to design materials with optimal features and improved performances. This article describes green synthesis concepts developed to prepare carbon materials with tuned porosity, structure, functional groups and embedded metallic nanoparticles. By carefully chosen examples, the impact of such carbon characteristics, independently tuned one from each other, on the performances in energy storage devices (supercapacitors, batteries and hydrogen storage) is highlighted *via* novel established correlations.

**Keywords** Carbon, hybrid carbon, porous carbon, hard carbon, supercapacitor, battery, hydrogen storage.

**Résumé** **Matériaux carbonés nanostructurés pour des applications dans le stockage de l'énergie**  
Les matériaux carbonés et carbonés hybrides nanostructurés font l'objet d'une attention particulière dans de nombreux domaines d'application tels que le stockage de l'énergie, l'adsorption ou la catalyse. Le développement de procédés de synthèse écoresponsables, simples et peu onéreux permettant le contrôle fin des caractéristiques du carbone représente un vrai défi dans l'objectif de concevoir des matériaux présentant des propriétés optimales pour des performances sans cesse améliorées. Cet article décrit des procédés verts développés pour préparer des matériaux carbonés intégrant des nanoparticules et offrant porosité, structure et chimie de surface contrôlées. À travers des exemples soigneusement choisis, l'impact de telles caractéristiques du matériau, ajustées finement et indépendamment les unes des autres, sur les performances dans des dispositifs de stockage de l'énergie (supercondensateurs, batteries et stockage de l'hydrogène) est mis en évidence *via* l'établissement de corrélations inédites.

**Mots-clés** Carbone, carbone hybride, carbone poreux, carbone dur, supercondensateur, batterie, stockage de l'hydrogène.

Carbon nanostructured materials are versatile and widespread in many fields of applications such as energy storage (batteries, supercapacitors, fuel cells), environmental applications (water and air purification/remediation) and catalysis. In all these applications, the performances are closely related to the material characteristics, hence, particular tuneable carbon features are required. Commercially available carbons lack of control over their properties is the reason why it is important to develop new synthetic pathways affording materials with controlled features. The implementation of sustainable concepts (green and renewable precursors/solvents) along to simpler and less time and energy consuming processes to obtain these materials is of great importance in view of developing more environmentally and cost-effective manufactory. This article will therefore present several achievements obtained in this regard.

Moreover, the article will present an overview on how the carbon material characteristics can be modulated *via* the utilization of these novel developed synthesis routes. The control of carbon porosity, structure and surface functionalities are of prime importance in many applications since they may impact the final performances. To improve the performances of carbon materials and to enlarge their application range, the association with metal-based nanoparticles (NPs) is a new required tendency. In such case, the control of NPs size, composition and location is researched and several ways to achieve this are presented along with some examples.

By carefully chosen examples, we will try to highlight how carbon and carbon hybrid material characteristics influence the performances when they are used as electrodes in supercapacitors, batteries or for hydrogen storage, although we have used these materials as well like absorbents for

liquid/gas molecules, in catalysis or medical applications. Several obtained properties-performances correlations will guideline for better material selection for a particular application. The results of this works may open new doors for the fabrications of sustainable carbon-based materials with optimal features for a broad range of applications.

#### How to design carbon materials with tuned porosity?

Carbon porosity (the specific surface area, the pore size/geometry and the pore volume) is one of the main important characteristic having a great impact in many applications. The specific surface area may influence the adsorption capacity of a liquid or gas molecule (pollutant molecules from water/air, electrolyte ions in a supercapacitor...) or the irreversible capacity of the first charge/discharge cycle in a battery. On the other hand, the pore size may limit the access of the molecules through the materials, therefore they must have an optimal size in order to allow efficient adsorption process. Usually, carbon materials with various surface area and pore size are commercially available and industrially used for several applications (water cleaning, as gas filters...). However, in such materials the pore size/geometry is random which make the utilisation limited for target applications and also for fundamental studies aiming to understand the correlations between the carbon characteristics and their performances in different applications.

To overcome this lack of porosity control, researchers developed specific synthesis pathways affording the preparation of carbon materials with ordered monodispersed pores, precise geometry and in addition with connectivity

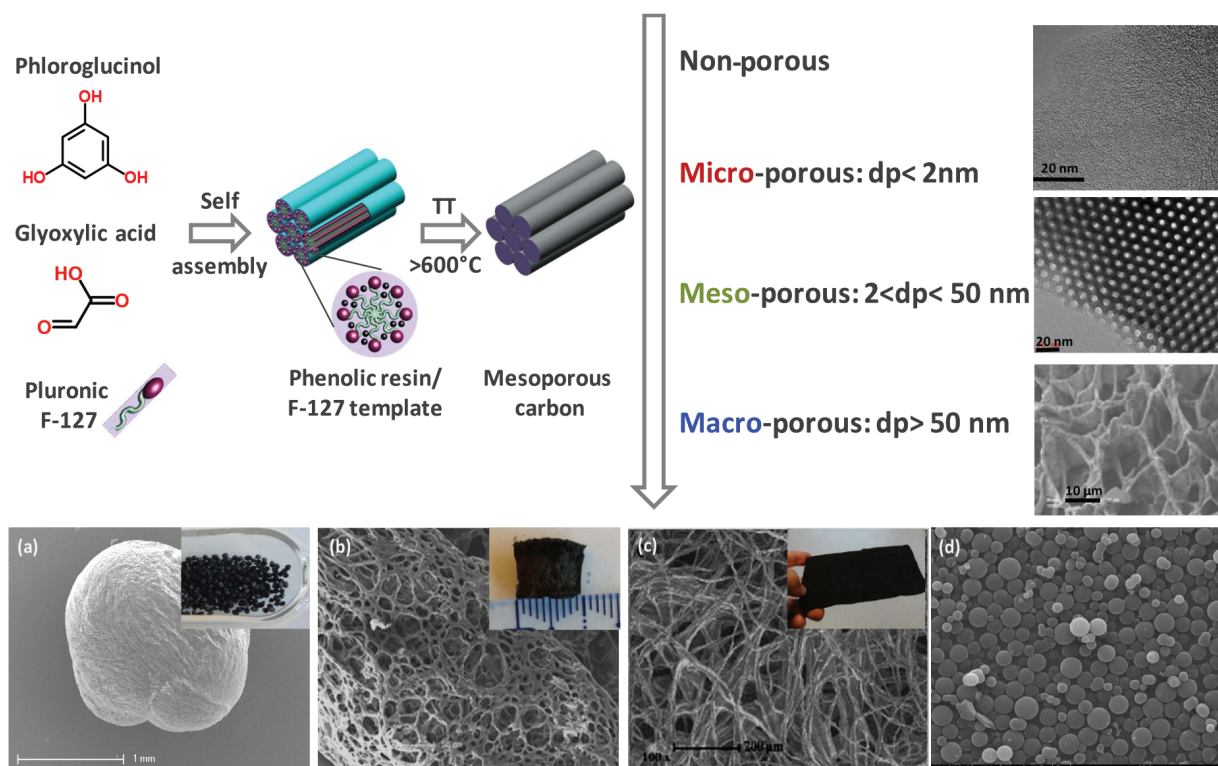


Figure 1 - Left: synthesis schema of a mesoporous carbon by soft-template route using phenolic resin as carbon source and Pluronic F-127 as template (pore and structure directing agent) [2]. Right: types of carbon materials that can be obtained by using this approach; TEM image of: non-porous carbon obtained in the absence of the template at high temperatures ( $> 1100^{\circ}\text{C}$ ) [4]; microporous carbon obtained by activation process [3]; macroporous carbon obtained by freezing-drying process of the chitosan resin/F-127 assembly [5]. Down: morphologies of carbon materials: (a) carbon beads [5]; (b) carbon monolith; (c) carbon fibre filters [6] and (d) carbon spheres [7].

between the pores *via* channels providing thus fast diffusion, uniform processes inside the pores and convenient nanoreactors for molecule confinement. However, only two synthesis pathways generate such materials with ordered porosity, and the so called “soft-template” route becomes in the recent years more popular than the “hard-template” one due to the reduced synthesis steps, less dangerous chemicals involved and flexibility. A “soft-template” designates a triblock polymer able herein to self-assemble with a phenolic resin in ordered nano-assemblies which by thermal treatment at high temperature ( $> 600^{\circ}\text{C}$ ) decompose to form mesopores in the carbon network derived from the phenolic resin decomposition (*figure 1*). In most of the previous published works ( $\sim 2500$ ) [1], phenol-formaldehyde resins are used as carbon source due to their high ability to self-assemble, high carbon yield and possibility to be shaped in different forms. However, they are highly toxic for humans and environment. In our works, we firstly proposed a green substituent for formaldehyde, i.e. glyoxylic acid extracted from plants [2], which contains beside the aldehyde group a carboxylic group able to catalyse the polymerization reaction between phloroglucinol-glyoxylic acid. In such case, a strong acid/base and a heating step usually required in the case of phenol-formaldehyde system was no longer necessary. The pore size/geometry control can be achieved by changing the ratio between the precursors, i.e. phloroglucinol/glyoxylic acid/Pluronic F-127 (soft-template). Thus, carbon materials with mesopore size between 3 and 14 nm and hexagonal, cubic or worm-like architecture could be obtained (*figure 1* right shows a carbon material with an ordered hexagonal pore arrangement). Other parameters such as the way of casting the precursor solution, the drying temperature or the aging time impacted the porosity of the prepared carbons.

Starting with this pioneer work [2], several approaches have been developed in order to vary the porosity and the morphology of the carbons, and also to reduce the environmental and energy impact of the synthesis routes for large scale preparation purpose. Few examples regarding these aspects will be given in the following part.

For many applications, the development of specific surface area is very important to allow high adsorption capacities. The specific surface area of carbon materials obtained by the soft-template route is ranged from  $500$  to  $700\text{ m}^2/\text{g}$ , which need to be improved in order to meet the requirements of practical applications. Activation route at high temperature with gases ( $\text{CO}_2$ ) or chemicals ( $\text{KOH}$ ) is one of the main known route to achieve better surface area, but in such case the consumption of carbon occurs, hence the recovered final carbon amount is low. In our research, we explored cheap alkaline salts ( $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{LiCl}$ ) which allowed to preserve the carbon yield and in addition could be recovered in the end of the reactions. Depending of the metal salt type and the reaction conditions, highly microporous carbons (pore size  $< 2\text{ nm}$ ) and high surface area up to  $1700\text{ m}^2/\text{g}$  [3] were obtained, which moreover present unusual graphitic-like structure, therefore good electronic conductivity. Such materials combining high surface area and graphitic structure are currently explored as electrodes for supercapacitors.

On the contrary, in batteries applications, the presence of porosity is not desired since it induces the electrolyte decomposition with the formation of a solid electrolyte interphase (SEI) which participates to the increase of the irreversible capacity during the first charge/discharge cycle of the battery. To obtain non-porous carbon using green phenolic resins, we developed a very easy and rapid route where only phloroglucinol and glyoxylic acid are used in water

(no template) resulting a phenolic resin gel which treated at high temperature ( $> 1100^{\circ}\text{C}$ ) gives non-porous disorder carbons (hard carbons) which were able to deliver a high electrochemical capacity (270 mAh/g) when employed as electrode in sodium-ion batteries [4].

Combining several types of porosity, i.e. microporosity (pore size  $< 2$  nm), mesoporosity ( $2\text{ nm} < \text{pore size} < 50$  nm) and macroporosity (pore size  $> 50$  nm) is for some applications of great importance since each class of pores may contribute to different processes, i.e. micropores/mesopores to ion adsorption and mesopores/macropores to ion diffusion. By combining the soft-template route with a freezing-drying approach, carbon materials with hierarchical porosity were prepared (*figure 1* right) using chitosan biopolymer as carbon source [5]. These materials can be shaped in form of spherical beads ( $\sim 1$  mm size) or monoliths ( $\sim 1$  cm) and they can be easily handled and recovered from reaction medium in practical applications as compared to power-like carbons (*figure 1* down, a, b). By combining the phenolic resin with cotton waste fabrics, carbon fibre filters of large size (few cm) could be obtained (*figure 1* down, c) and used as adsorbents for dye removal from water [6]. Another way to change the carbon morphology is to use supplementary molecules in the synthesis; this is the case of triethylenediamine (TEDA) that we proposed for the first time and was able to catalyse and cross-link the phenolic resin resulting in homogenous carbon microspheres ( $5\text{ }\mu\text{m}$  size, *figure 1* down, d). The proposed TEDA herein is very advantageous since it allows to perform the synthesis at room temperature in water as solvent and readily easily to up-scale [7] contrary to most of carbon spheres syntheses using toxic phenol-formaldehyde and strong-smelling corrosive ammonia under hydrothermal conditions (high temperature and pressure), and therefore not suitable for industrial applications. In few other works, we explored the light irradiation for the synthesis of carbon materials which allowed advantageously to decrease the reaction time, to remove some synthesis steps and even to directly carbonise the polymer resin [8].

Therefore, we could see that many parameters can be varied in order to optimise the porous structure and the morphology of carbon materials. As demonstrated, this control of properties can be done in addition *via* novel eco-friendly synthesis pathways. The understanding of the involved synthesis mechanism is of key importance for designing new materials and was studied in detail in several herein cited works.

It is worth to mention that for obtaining correlations between one specific carbon characteristic and the performances, one of the great synthesis challenges is to modify only the desired characteristic without modifying the others which may also contribute to the performance. Only a such precise control on the material synthesis may allow to get new insights in the material structure-properties correlations. This is the case of the example provided herein obtained in the frame of a collaboration with CRMD (Centre de Recherche sur la Matière Divisée) and PCM2E (Physico-Chimie des Matériaux et des Électrolytes pour l'Énergie), research groups specialized in supercapacitor devices and electrolyte design, where we could find a correlation between the pore size of the carbon and the capacitance of a supercapacitor (*figure 2a*) working in lithium-based electrolyte. The optimal carbon pore size to achieve high capacitance in organic electrolytes (tetraethyl ammonium, tetrafluoroborate  $\text{TEABF}_4$ ) in acetonitrile (ACN) solvent was determined in the literature to be  $0.7\text{ nm}$  [9-10], size corresponding to the desolvated electrolyte ions. However, the influence of lithiated electrolytes (bis(trifluoromethane)sulfonylimide, LiTFSI) was never studied before. By preparing a family of carbon materials with tuned pore size and similar surface chemistry, we could show that the optimal pore size for a lithium-based salt (LiTFSI) dissolved in EC/PC/3DMC solvent is much higher, i.e.  $1.1\text{ nm}$  (*figure 2b*) [11]. Surprisingly, when the LiTFSI was dissolved in ACN solvent, the pore size was the same as for  $\text{TEABF}_4/\text{ACN}$  ( $0.7\text{ nm}$ ), suggesting a strong effect of the solvent as well on the capacitance (*figure 2b*). It was found that the solvent properties (polarizability, dipole moment) induce strong interactions between the electrolyte ions (in the case of EC/PC/3DMC), leading to solvation shells difficult to be removed before entering in the carbon pores, therefore larger pore size was required. Therefore, for achieving the highest capacitance, the carbon pore size must be matched with both the electrolyte and solvent size.

### Playing with carbon surface functionalities and defects

The carbon surface chemistry (nature and amount of functional groups) is another key parameter to be controlled since it influences the hydrophilic/hydrophobic behaviour of carbon and in consequence the interactions with the liquid or gas molecules impacting therefore the performances. Most carbon

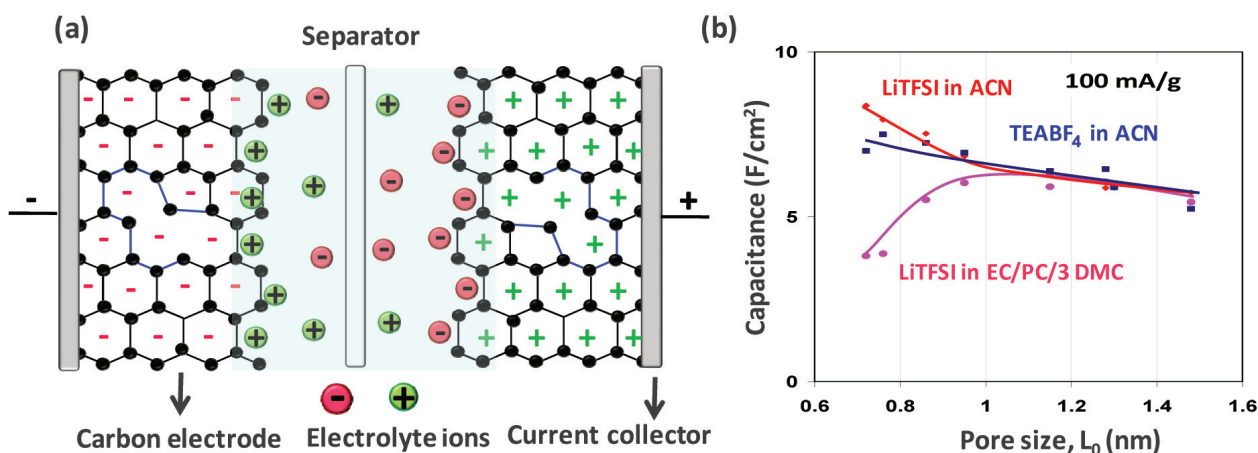


Figure 2 - (a) Schematic representation of an electrochemical double layer capacitor; (b) relation between the carbon specific capacitance normalized by specific surface area and the pore size in LiTFSI in EC/PC/3 DMC,  $\text{TEABF}_4$  in ACN and LiTFSI in ACN electrolytes [11].



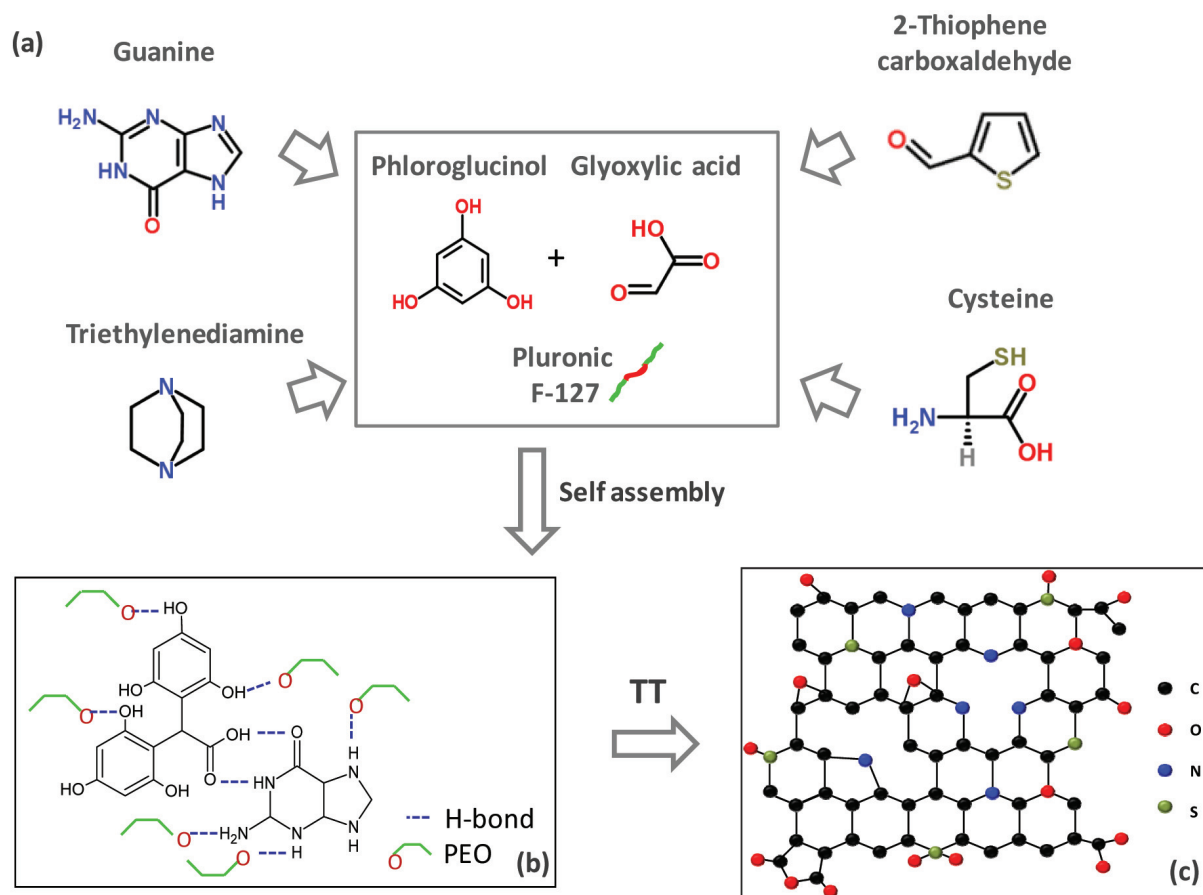


Figure 3 - Schematic representation of the direct synthesis of heteroatom-doped carbon materials: (a) general synthesis pathway including few examples of molecules able to provide heteroatoms in the carbon skeleton [7, 13-14]; (b) example of interaction of phenolic resin with a dopant molecule (guanine) and the Pluronic F-127 template [14]; (c) schema of a carbon skeleton containing N, S and O heteroatoms and functional groups.

precursors naturally contain oxygen heteroatoms in their structure; therefore, the resulting carbon materials present oxygen functional groups such as carboxylic, phenol, ether, hydroxyl in their surface. The amount/nature of such oxygen groups depends on the initial precursor chemical composition but also of the pyrolysis temperature used to prepare the carbon. Increasing the temperature up to 900°C under inert or reducing atmosphere (Ar, H<sub>2</sub>) leads to the complete removal of the oxygen functional groups and more hydrophobic carbons. On the other hand, the modification or enrichment of the surface chemistry is often desired in order to favour certain interactions and/or to improve the adsorption efficiency and specific post-synthesis thermal or chemical treatments using oxidising agents (O<sub>2</sub>, H<sub>2</sub>O<sub>2</sub>, HNO<sub>3</sub>...) can be performed in this aim [12]. Other heteroatoms such as nitrogen or sulphur are introduced in the carbon structure to alter the electronic structure or the basicity of carbon by using post-thermal treatment under gases such as NH<sub>3</sub> or H<sub>2</sub>S at high temperatures (> 600°C). An alternative and convenient way is to directly (one-step reaction) dope the carbon with N and S by incorporating in the reaction medium molecules containing these heteroatoms. For instance, we have investigated several novel molecules, i.e. guanine, triethylenediamine, thiophene carboxaldehyde or cysteine in the soft-template synthesis (figure 3a) and carbon material having two or three heteroatoms in the structure could be obtained (figure 3c). The incorporation of N, S heteroatoms in the carbon structure is possible due to some complex mechanisms which involve either the cross-linking of these molecules to the phenolic resin one, the self-assembly with the template or weak H-bonding

with the phenolic resin as determined by <sup>13</sup>C NMR analyses and schematically represented in figure 3b.

While the nature of functional groups may be assessed by several classical methods, the quantification of functional groups in whole carbon material can be accurately determined by temperature programmed desorption coupled with mass spectrometry (TPD-MS). This technique developed in our laboratory allows also to quantify the defects contained in the carbon materials. Carbon defects are mainly located in the edge planes of carbon and constitute the so-called active sites or active surface area (ASA). The ASA is a key parameter which was found to influence the irreversible capacity of graphite in Li-ion batteries. However, the influence of carbon surface chemistry, active surface area and porosity on the Na-ion batteries were not well known. Such Na-ion batteries (figure 4a) are aiming to replace the Li-ion batteries due to the lack of Li in the nature in the near future. The mechanisms of Na storage in carbon is still a controversial subject in the literature and our recent studies shed some light in this regard [15-16]. We could establish clear relationship between the irreversible/reversible capacity and the hard carbon properties [15] by using carbon synthesized by simple annealing of cheap, eco-friendly and abundant cellulose at different temperatures (figure 4b). The irreversible (sloping) capacity was found to decrease with the decrease of the porosity, functional groups and active surface area while for the reversible (plateau) capacity an opposite behaviour was observed (figure 4b). Therefore, we could show that the reversible storage occurs *via* Na<sup>+</sup> intercalation between the pseudo-graphitic carbon planes while the irreversible Na<sup>+</sup> storage/adsorption takes place mainly in the

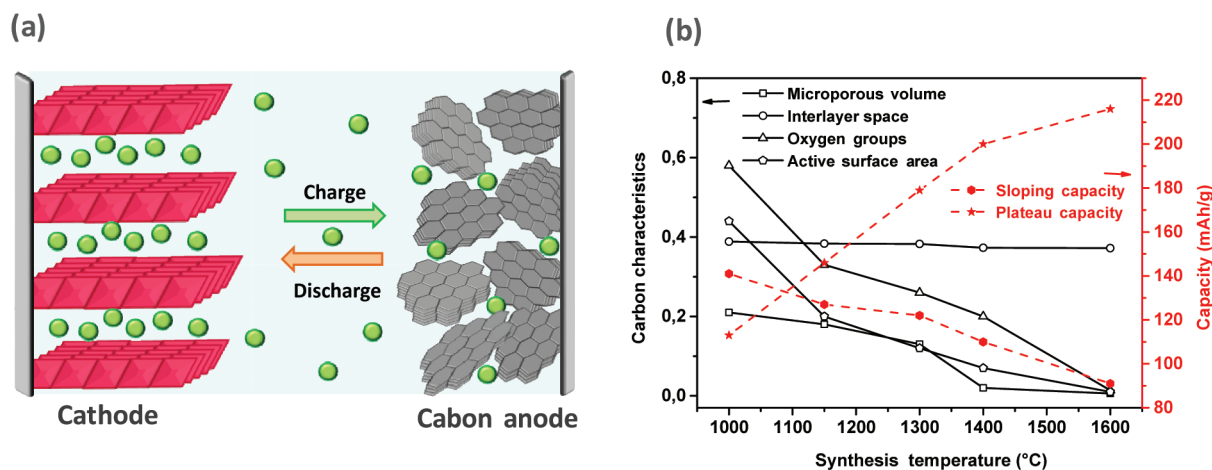


Figure 4 - Schematic representation of a Na-ion battery (a); correlation between the characteristics of a hard carbon (porosity, surface chemistry and structure) and the electrochemical capacity in a Na-ion battery (b) [15].

carbon porosity and defects. For carbon materials heat-treated at higher temperatures than 2000°C, we could firstly observe only one single plateau (mechanism) for Na<sup>+</sup> insertion in carbon [16].

This fundamental research performed in the frame of the European project Horizon 2020 (Naiades) and the RS2E network (Réseau sur le Stockage Electrochimique de l'Énergie) was accompanied by an important technological achievement, i.e. the development of the first Na-ion battery prototype using hard carbon as anode.

### Carbon materials containing confined metallic-based nanoparticles

The association of carbon materials with metal-based nanoparticles (NPs) (metal, metal oxide, metal nitride...) allows to extend the range of applications of carbon materials and also to improve their performances *via* synergetic effects. The carbon network as support for NPs may provide higher surface area, better electronic conductivity, improved mechanical properties and thermal/chemical resistance. Particularly, the NPs of very small size are of great interest since they present rather different properties/performances as compared to their bulk counterpart. To take benefit of these advantages, it is essential to synthesize metal NPs with small and dispersed size. However, the synthesis and stabilisation of such small NPs remains still a challenge. In this aim, carbon materials with high surface area and uniform pores proved to be ideal scaffolds to confine metal nanoparticles, limiting therefore their growth and allowing the formation of small size and well dispersed particles.

Generally, the most spread synthesis approach relies on the impregnation of the carbon with a solution of metallic precursors followed by a thermal or chemical reduction step (figure 5). Depending of several factors that must be carefully optimised (carbon support type and characteristics, synthesis temperature, metal salt nature...), small particles below 5 nm can be obtained. In addition to this indirect approach consisting in the preparation of carbon support firstly and secondly of the NPs, we developed as well direct (one-pot) synthesis routes allowing simultaneous formation of carbon and NPs by simply mixing the carbon and metal salt precursor solutions followed by thermal annealing (figure 5, top).

The advantage of such approach is not only related with the reduction of the synthesis time and steps but also to

the fact that no chemicals/reducing gases are required to produce the particles, the carbon playing a role of reducing agent during the thermal annealing. Despite the simple synthesis approach from practical point of view, the control of carbon characteristics and NPs is much difficult to achieve since the properties of carbon precursors may influence the NPs formation and *vice versa*, the properties of metal salt may modify the carbon characteristics. Different types of metal-based particles have been prepared for the first time by this approach with a uniform distribution in the carbon network (figure 5, down). However, in such case, the size of the NPs is usually higher than those obtained by indirect route and mainly related to the higher annealing temperature (> 600°C) required for the formation of the carbon network.

These two synthesis approaches which could be also assisted by laser or by microwave in our works (figure 5, top) in order to diminish the synthesis time and to afford other material properties are very flexible and efficient to tune the metal particle size and dispersion, their composition and location in the carbon network. For instance, the particles prepared by one-pot are located in the carbon walls or in the carbon surface while for the indirect impregnation route they are more likely located in the carbon pores or in the surface of carbon. These modifications of the NPs size, composition and location may induce different material performances as we will highlight it by two detailed examples.

For instance, the precise controlled of Pd-based nano-alloy NPs characteristics allowed to explore fundamental interactions with the hydrogen at nanometer scale in the frame of the ANR Genesis project performed in close collaboration with the Institut de Chimie et des Matériaux Paris-Est (ICMPE-Thiais). It was established that the hydrogen storage capacity is influenced both by the particle size and by the composition [19]. For instance, when Pd is substituted with 10 or 25 at.% of Co, the hydrogen absorbed per metal unit (H/M) decreases (figure 6a). If the substituted amount is larger than 25 at.%, the hydrogen absorption is suppressed no matter the type of substitution metal used (Rh, Ni, Au, Ir...). In the case of Pd<sub>90</sub>Co<sub>10</sub>, the Pd hydride could be formed in a similar manner as for pure Pd, while in case of Pd<sub>75</sub>Co<sub>25</sub> only hydrogen adsorption on particle surface occurred. For these compounds, the evolution of the H/M report is different as well, it increases with the increase of the particle size for Pd and Pd<sub>90</sub>Co<sub>10</sub> while an opposite behaviour is observed for Pd<sub>75</sub>Co<sub>25</sub>.

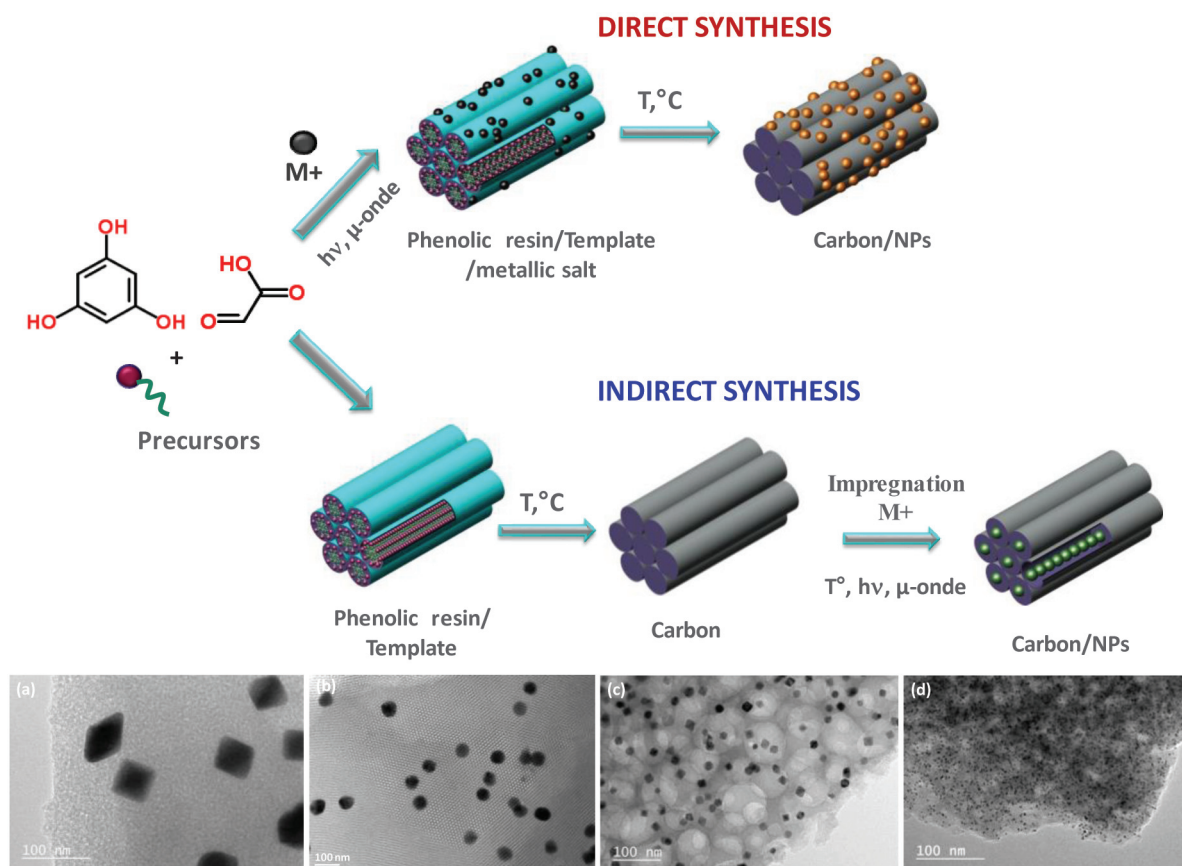


Figure 5 - Top: synthesis pathways used to prepare carbon hybrid materials (carbon containing metal nanoparticles (NPs): direct one-pot route involving simple mixture of carbon and metal precursors with the template followed by thermal annealing [17]; indirect impregnation route implying the preparation of the carbon support in a first step followed by its impregnation with a metallic salt solution and a reduction step [18]. Bottom: TEM images of carbon materials decorated with metal nanoparticles: (a) Pd-Pt nanoalloy, (b) Ni-Co alloy, (c) Pd and (d) Rh.

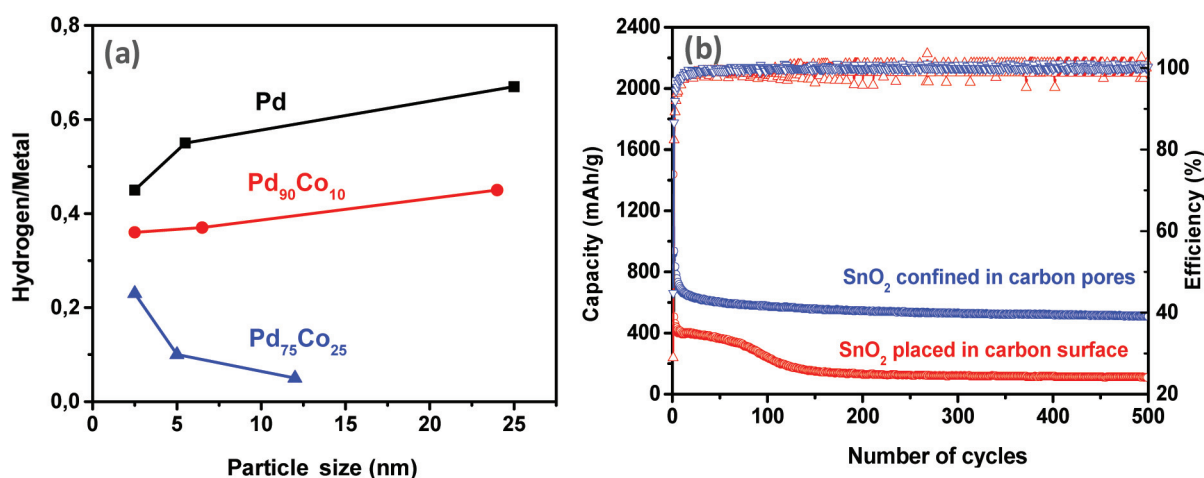


Figure 6 - Correlation between the hydrogen absorbed per metal unit vs. the Pd-Co particle size and composition [19] (a) and electrochemical capacity in Li-ion battery of carbon/SnO<sub>2</sub> hybrid materials (two cases: SnO<sub>2</sub> nanoparticles confined and unconfined in carbon pores) (b) [20].

In other works, we could highlight for the first time in a joint work with ICGM (Institut Charles Gerhardt, Montpellier) the importance of the nanoparticle location in the carbon network in order to limit their volume expansion, their agglomeration, and to provide long cycle life when used as anodes in Li and Na-ion batteries. For example, SnO<sub>2</sub> nanoparticles (~2 nm) could be confined in the pores of a mesoporous carbon and it exhibited higher capacity and long term cycle life (500 cycles) without loss of capacity (figure 6b), contrary to a composite where the SnO<sub>2</sub> NPs of same size is placed in the carbon surface showing lower capacitance and rapid fading after only 100 cycles [20]. The electrochemical performances of such mesoporous carbon with confined SnO<sub>2</sub> NPs was remarkable

particularly in terms of stability and time life and was possible due to limitation of particle growth during the repeated charge/discharge cycles.

### The future of carbon materials prepared by "green" concepts

Carbon and carbon hybrid materials can be designed with optimal characteristics for applications mainly in the field of energy storage, adsorption and catalysis. One original aspect highlighted is the development of environmentally friendly synthesis pathways involving green precursors and solvents, low temperature, reduced steps and easy preparation



for up-scale. Many of these materials were successfully used in several applications; however, herein we preferred to present few key examples which allow to understand the importance of carbon characteristics on their performances in supercapacitors, batteries and hydrogen storage. Carefully tuning a targeted carbon feature independently from others allows to decouple concomitant effects on the performances and to establish precious relationships between the carbon characteristics (porosity, surface chemistry and defects) and their performances. Although most of the materials presented herein are explored yet only at laboratory scale due to their high price induced by the precursors and the manufacturing process, they allow on one hand to fundamentally understand which are the optimal carbon characteristics for improved performances. On the other hand, taking into consideration the increase interest in environmental aspects in nowadays, we can hope that some carbon materials prepared by "green" concepts will become in the future valuable materials for up-scaling and utilisation in various real-life applications.

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#### Camélia MATEI GHIMBEU,

chargée de recherche CNRS à l'Institut de Science des Matériaux de Mulhouse (IS2M)\*, membre du Réseau sur le Stockage Electrochimique de l'Energie (RS2E). Elle a reçu la Médaille de bronze du CNRS en 2017.



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\*Université de Strasbourg, Université de Haute-Alsace, Institut de Science des Matériaux de Mulhouse, UMR 7361 CNRS, 15 rue Jean Starcky, BP 2488, 68057 Mulhouse Cedex.  
E-mail: camelia.ghimbeu@uha.fr

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