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# Chemistry in all its States

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## EDITOR'S NOTE

Source: *La lettre*, no. 33, May 2012

### 1 • The Microwave Oven: from the Kitchen to Synthesis Chemistry

- 1 “A microwave oven is a domestic appliance that is mainly used to heat up food quickly, by agitating the water molecules contained in the food under the effect of a microwave radiation” (Wikipedia). From the outset, this technology was mainly used in the kitchen and sometimes in highly precise industrial applications. However, for the last twenty years or so microwave has also been used in chemistry laboratories, to activate chemical reactions and to prepare “advanced” materials of great value for new technology.
- 2 In order to understand properly the mechanisms of microwave action, we first have to consider the fundamental concepts of interaction between electromagnetic waves and matter, particularly with water molecules, followed by their energetic transformation in molecular rotational movements and the heating up of the immediate environment of those same molecules. That is how microwave-assisted reactions lead to a significant reduction of the reaction time. Moreover, the yield and even the selectivity of reactions are often improved. The application of microwave saves considerable time and energy (Green Chemistry). Microwave activation can be applied for organic synthesis in the so-called “dry media” conditions, that is, without solvents, where finely divided inorganic solids act as a solvent in a sense, while also supporting the reactants. What initially seemed to be a chemical synthesis revolution is nowadays being used for a wide variety of applications, not only in laboratories but also in certain industrial processes. In fact,

some current uses offer interesting possibilities of synthesis in both organic and inorganic synthesis chemistry, for:

- 3 - solid-state reactions
- 4 - preparation of ceramic materials
- 5 - hydrothermal synthesis
- 6 - digestion of solid materials for chemical analysis
- 7 - polymerization of monomers and preparation of hybrid materials and composites, etc.

## 2 • Clays: a Raw Material for Advanced Materials

- 8 Amongst the most abundant, universal and accessible raw materials, clay is without a doubt a particularly interesting example. In fact, clays are used in widely diverse sectors of application, from the most traditional ones, like pottery and construction (bricks and tiles) to sophisticated materials like certain heterogeneous catalysts. This goes to show the real utility and great versatility of this family of silicates.
- 9 A raw material as simple as a clay can actually also have applications in domains associated with “advanced” technology. Clays can thus be transformed into materials for very specific purposes, or even into functional nano-materials. In order to illustrate these concepts, let us consider various examples constituting a concise review of clay-based functional materials, selected from research carried out within our research group over the last few years. At present, these clay-based materials are highly valuable for various applications, such as special adsorbents, specific catalysts, anti-pollution materials, components of electrical and electrochemical devices, selective membranes, photo-active systems, or even in the preparation of next generation adjuvants for vaccines. Among the most recent examples, clays have served as the base for the preparation of new multi-functional hybrid materials such as magnetic adsorbents or supported graphenes. When treated by means of interaction with ferrofluids, lamellar clays, like montmorillonite, or fibrous ones, like sepiolite, are transformed into super-paramagnetic materials that preserve their capacity to absorb organic species and to exchange cations in solution. Afterwards, their super-paramagnetic character makes easier its capture using a magnetic field, which allows the application of those materials for the sequestration and elimination of polluting substances in water without resorting to complex recovery processes such as filtration, centrifuging or membrane processes. Furthermore, clay-supported graphene has been produced through the controlled carbonization of totally harmless organic compounds such as caramel or gelatine. This is a new process for the preparation of graphene-based materials using a “soft route” which leads to carbon-clay compounds with properties that are simultaneously characteristic of both components, that is, molecular adsorption and electric conductivity.

## 3 • The “Intracrystalline Laboratory”: The Chemistry in Confined Nano-Spaces

- 10 Certain nanoporous microcrystalline solids provide spaces where molecular access is controlled by different factors, mainly steric and topochemical ones. The size and

shape, as well as the chemical nature of the guest molecules play a determining role in allowing their passage through the nanowindows, in their arrangement inside the solids and, in some cases, in the event of unusual chemical transformations within the solid. Cavities inside solids with a 3D organisation, typically zeolites and related materials, the inter-lamellar spaces of solids with a 2D organization or the tunnels of monodimensional solids (1D), like certain mesoporous silicas, carbon nanotubes and fibrous clays, are all nanometric domains that offer the possibility of implementing a different chemistry to that which is produced in conventional homogeneous environments. These are real intracrystalline laboratories in which, for instance, water molecules can find themselves in an abnormal state of dissociation, typically 1,000 times greater than in water in a normal state. This leads to very high acidity in these environments, thanks to which unexpected catalytic transformations on the surface of these solids can be induced. Selected examples are the adsorption and the selective transformations in nanoporous solids related to ZSM-5 zeolite in the alkylation of toluene, molecular transposition reactions in smectite-type clays, mesoporous silicas asymmetrically grafted through organic functions and carbon nanotubes that can encapsulate nano-crystals with various chemical compositions. Regarding solids with a monodimensional nanoporous organization, we can also consider the example of the pigment known as Maya Blue, made by the ancient Mayans. In this pigment, the indigo dye was encapsulated at molecular level within the structural nanospaces of a microfibrillar clay called palygorskite. Owing to this “encapsulation” in confined spaces, the characteristic blue colour lasted for centuries. Actually, this is the result of a nanotechnology developed about ten centuries ago!

## 4 • Biohybrids, a New Type of Materials at the Interface of the Inorganic and Living Worlds

- 11 The combination on a nanometric scale of compounds originating from biological species with inorganic solids produces a class of nanostructured materials called biohybrids. These materials represent a very important progress in the field of functional materials, as the incorporation of biological entities such as cellular fragments or even entire micro-species confers properties to the involved inorganic solids which by far surpass those of systems that exclusively use synthetic compounds. In the preparation of biohybrids by means of bottom-up methods usually applied to nanotechnology, the scientist uses construction units that are well-defined in soft chemistry preparation conditions. This method is very useful as it prevents the alteration of biological entities that are generally fragile and sensitive
- 12 In this respect, it is worth mentioning several systems that are of particular interest, like the immobilization of enzymes by sol-gel matrices, the inclusion of chlorophyll in mesoporous silicas, the encapsulation of living cells in rigid or flexible matrices, and the intercalation of biopolymers in solids with a 2D organization. Among biohybrids, those resulting from the assembly of inorganic solids with biopolymers have been called bionanocomposites. Certain bionanocomposites are of natural origin, as it is the case of bone or pearl, consisting of phosphate or carbonate particles bound on a nanometric scale with proteins, for example collagen or lustrin A. The use of various inorganic solids, such as silicas and silicates, combined with the wide range of choice offered by biopolymers, opens up to limitless possibilities for engineering and synthesis

of new biomimetic materials. For example, lamellar or fibrous clay silicates combined with biopolymers were recently used to develop new biohybrids elaborated in the form of films or foams, useful as membranes for separation of gases or as ultra-low density materials with cellular structure. Furthermore, these materials are biodegradable, biocompatible and fireproof, which leads to multiple applications in a wide variety of domains, for instance acoustic and thermal insulation. Biomimetic membranes based on biohybrid systems may also constitute an adequate environment to immobilize enzymes, microalgae cells and viral particles. These systems, which are halfway between the inorganic and living worlds, display interesting bioactivity properties and are currently under study for applications in domains pertaining to the production of biomass, vaccines or highly selective biosensors.

• Lectures online at [www.college-de-france.fr](http://www.college-de-france.fr)

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## AUTHOR

### EDUARDO RUIZ-HITZKY

is, since 1988, a professor at the CSIC (National Research Council of Spain), and is currently the Head of the Hybrid, Biohybrid and Porous Nanostructured Materials team at the Materials Science Institute of Madrid. He has spent several decades doing pioneer research on hybrid and biohybrids materials, especially those involving derivatives of particulated inorganic solids such as clay minerals.