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## Spray synthesis of nanomaterials for catalysis, a wind of change

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**Electronic version**

URL: <https://journals.openedition.org/lettre-cdf/765>  
DOI: 10.4000/lettre-cdf.765  
ISSN: 2109-9219

**This article is a translation of:**

Nanomatériaux pour la catalyse : le spray a le vent en poupe - URL : <https://journals.openedition.org/lettre-cdf/120> [fr]

**Publisher**

Collège de France

**Printed version**

Date of publication: 1 June 2009  
Number of pages: 31-32  
ISSN: 1628-2329

**Electronic reference**

Clément Sanchez, "Spray synthesis of nanomaterials for catalysis, a wind of change", *La lettre du Collège de France* [Online], 4 | 2008-2009, Online since 15 November 2010, connection on 17 August 2022. URL: <http://journals.openedition.org/lettre-cdf/765> ; DOI: <https://doi.org/10.4000/lettre-cdf.765>

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### Spray synthesis of nanomaterials for catalysis, a wind of change

Aluminosilicate zeolites are microporous crystals used as heterogeneous catalysts for petroleum transformation. Most part of time their porous networks present microporous restrictions of size below 1 nm, which limits their application to small molecules. Since their first industrial application 45 years ago, an alternative low-cost material presenting similar acidic properties, high surface area and larger pores is needed for using at the best the huge volume of heavy oil available made of high molecular weight molecules. A first part of the problem was solved in 1992 with the first preparation of mesostructured aluminosilicate materials presenting periodically organised pores at mesoscale.

Although, their amorphous nature reduced their potential activity, as a result of their moderate acidity, and hydrothermal resistance. A solution to this problem was found by a research team of the Laboratoire de Chimie de la Matière Condensée de Paris led by Clément Sanchez (UPMC/Université Pierre et Marie Curie-CNRS-Collège de France), in collaboration with researchers of the Institut Français du Pétrole.

By coupling sol-gel chemistry with a very low-cost and environmentally benign aerosol shaping process, LCMCP researchers successfully produced spherical submicronic particles which internal porous cavities are

at the same time tunable between 4 and 50 nm and exhibits a very high Bronsted acidity. For synthesizing such materials named LAB (for Large pores Aluminosilicates prepared with Basic solutions), they use a spray generating a mist of micron size droplets containing silica and alumina molecular precursors, and two organic structuring agents. Upon heating, droplets solvents (mainly water and residual ethanol) are evaporated and non volatile organic and inorganic components spontaneously self-assemble and form in a few seconds a dry powder exhibiting a mesostructure with periodically organized organic and inorganic

nanodomains. This physico-chemical quench allows the "freezing" of materials with new chemical compositions in metastable states which are hardly achievable by the usual precipitation method that favors usually the thermodynamical products. In a second time, the calcination of organic agents allows producing micro and mesoporous catalysts microspheres exhibiting amorphous and very acidic walls.

At the difference of the classical precipitation pathway, aerosol process involves a very limited number of preparation steps, produces material continuously,

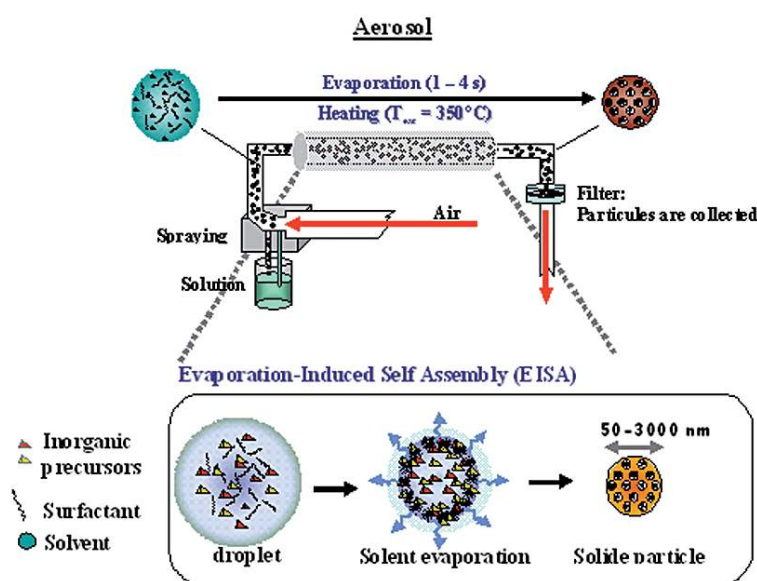


Figure 1: Scheme of the aerosol process used for the production of mesostructured LAB particles. The precursors solution is (i) sprayed to form micron-size droplets, (ii) droplets are dried in a hot zone for promoting their mesostructuration by EISA, (iii) dry particles are simply collected onto a filter.

allows a simple continuous collection of the powder and generates the strict minimum of wastes (vapors). The so-prepared catalysts exhibit exceptional catalytic activities. Moreover, these activities are maintained much longer than classical zeolites and need thus to be recycled less often. When one knows that it is possible to integrate within the porous structure some organic functions

and/or some functional nanoparticles, one understands that this aerosol / sol-gel coupling may give birth to a broad range of innovating catalysts with unexplored properties.

Finally, this strategy is not limited to the synthesis of catalysts but is already developed for the synthesis of new bioceramics and therapeutic vectors which hybrid organic/

inorganic structure allows integrating several collaborating functions such as enhanced contrast MRI imaging, hyperthermia and controlled drug delivery. Let's spray ! ■

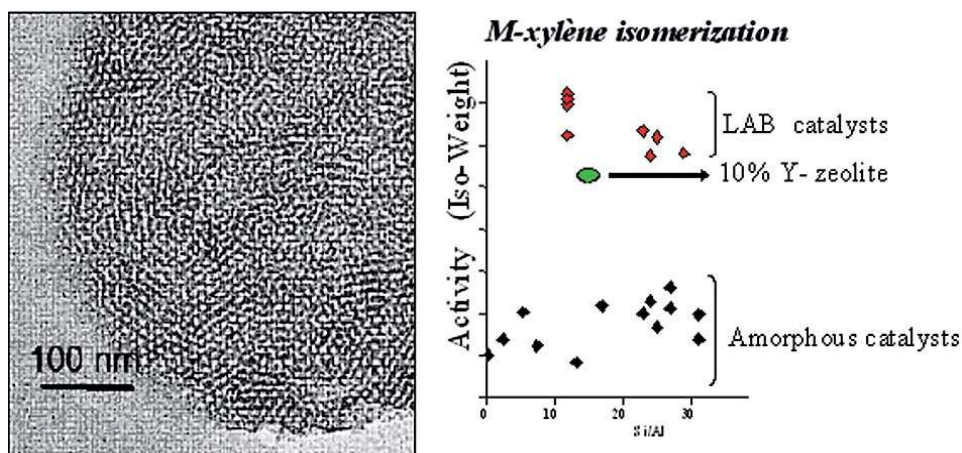


Figure 2: (left) TEM picture of one LAB particle. The porous network is evidenced by the periodical appearance of dark and white zones. (right) Graphic plotting the iso-weight activity of some LAB catalysts of different structures and chemical compositions. They are compared with the activities of industrial zeolitic and amorphous catalysts.

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