Characterization of nanocomposite porous catalysts

Maria Francesca Casula[1], Nicola Melis[1], Angelo Frongia[2], Maria Giorgia Cutrufello[2], Danilo Loche[3], Swapneel V. Thakkar [2,†], Maria Franca Sini [2], Gianmarco Sedda[2], Luca Pilia[1]

[1] Department of Mechanical, Chemical and Materials Engineering, University of Cagliari, 09123 Cagliari, Italy Email : mariaf.casula@unica.it
[2] Department of Chemical and Geological Sciences, University of Cagliari, 09042 Monserrato, Italy
[3]Nanostructures & Biotech Laboratory, Biological and Environmental Science and Engineering (BESE) Division, King Abdullah University of Science and Technology, Thuwal 23955-6900, Saudi Arabia
† Current address: Tronox, Research Center Thann, 95 Rue du Général de Gaulle, 68800 Thann, France.

Nanocomposites where a nanocrystalline (metal- or metal oxide-based) active phase supported on a matrix featuring extended porosity are of great interest for a wide range of applications, ranging from the development of biomaterials to innovative catalysts.

The functional properties and therefore the prospective areas of application depend on the different key features of the nanocomposites, such as: nanocrystal composition, size, and shape; nanocrystal distribution over the matrix and potential interactions; composition, surface area, pore structure and surface coverage of the matrix. This motivates the need for an extensive multi-technique characterization of the nanocomposites by combining structural, morphological, textural, and spectroscopic techniques.

Here, the characterization of nanocomposites based on mixed ferrite nanocrystals dispersed on a highly porous silica matrix, which has already demonstrated to act as an effective matrix for the design of catalysts with high stability, will be discussed.[1,2] The nanocomposites are obtained through sol-gel techniques followed by specific drying and thermal processing procedures in order to obtain the final porous solid.

X-ray powder diffraction (XRD), transmission and scanning electron microscopies (TEM, SEM), N_2 physisorption at 77 K, Fourier Transform Mid-Infrared spectroscopy (FT-IR), and microcalorimetry are used to characterize the porous nanocomposites. As the use of porous nanocomposites may contribute to develop sustainable and effective catalytic processes, the functional properties of the nanocomposites will be presented with specific reference to the catalytic production of bis- heterocyclic compounds which are found as natural metabolites and represent versatile intermediates for synthetic organic and pharmaceutical compounds [3], as depicted in the scheme in Figure 1.

In particular, the reaction of formation of a bis- indolyl methane (3,3′-((4-nitrophenyl)methylene)bis(1H-indole) in the presence of the investigated porous nanocomposites was studied by monitoring the reaction through ^{1}H NMR analysis of the crude mixture.[4] The nanocomposites were found to be active at room temperature for the investigated reaction, and this was associated to the surface, structural and textural features. Characterization of the nanocomposites after the catalytic reaction was also found to be relevant in showing the occurrence of trapped reaction species in the porous catalyst.

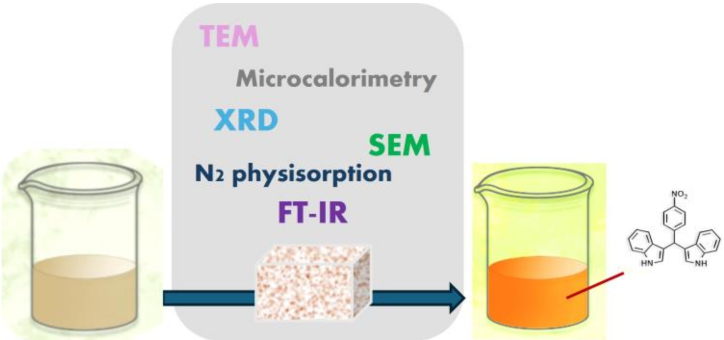

Figure 1 – Porous nanocomposites used as catalysts for the synthesis of a bis-indolyl methane derivative

REFERENCES

[1] Vanyorek L., Katona H., Loche D., Corrias A., Casula M.F., Konya Z., Kukovecz A., Kiricsi I. (2011) "Optimization of the catalytic chemical vapor deposition synthesis of multi-wall carbon nanotubes on Fe,Co(Ni)/SiO_2 aerogel catalysts by statistical design of experiments" *Journal of Physical Chemistry C*, 115, 5894-5902.

[2] Carta D., Montini T., Casula M.F., Monai M., Bullita S., Fornasiero P., Corrias A. (2017)"The Water Gas Shift Reaction over Pt-CeO_2 nanoparticles confined within mesoporous SBA-16" *J. Mater. Chem. A* 5, 20024-20034 .

[3] Grosso, C.; Cardoso, A.L.; Lemos, A.; Varela, J.; Rodrigues, M.J.; Custódio, L.; Barreira, L.; Pinho E Melo, T.M.V.D. (2015) "Novel Approach to Bis(Indolyl)Methanes: De Novo Synthesis of 1-Hydroxyiminomethyl Derivatives with Anti-Cancer Properties" *Eur. J. Med. Chem*., 93, 9–15.

[4] Melis N, Loche D., Thakkar S. V., Cutrufello M.G., Sini M.F., Sedda G., Pilia L., Frongia A., and Casula M.F.(2024) "Magnetic Aerogels for Room Temperature Catalytic Production of Bis(indolyl)methane derivatives", *Molecules* 29 (10), 2223