METALLIC AND BIMETALLIC NANOPARTICLES DISPERSED IN MESOPOROUS MCM-41

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In recent years, nanosized materials have received significant attention due to their impact in different processes and to their interesting physicochemical properties. Several diverse applications of these materials, e.g. high density magnetic recording, magnetic fluids, magnetic refrigeration as well as in photocatalysis, solar cells, photosensors, have triggered considerable research activities in the area nanotechnology [1-3]. Nevertheless, from the viewpoint of nanoparticles in confined environment, e.g. nanoparticles confined in micropores or mesoporous of certain molecular sieves [4] there is still a lot of work to be done. On the other hand, the discovery of MCM-41 mesoporous molecular sieves has allowed an immense scope for many researchers to work on these new type of materials, with uniform size and shape of the pores, arranged in different ways and with a very high surface area and adsorption capacity [5]. For such a reason, the purpose of this work is to synthesize and to characterize nanoparticles of Fe and Co and its corresponding bimetallic on MCM-41.

The mesoporous material MCM-41 (formed by a system of ordered channels), was synthesized hydrothermally according to a procedure outlined earlier [6]. The gel composition was $0.09SiO_2$: 0.02 Na_2O: 0.01CTAB: $40H_2O$. The metallic precursors were $Fe(NO_3)_3.9H_2O$ and $Co(NO_3)_2.6H_2O$. The nominal total metallic load for all the systems was 10 % atomic. The impregnation of the mesoporous materials was carried out following the conventional method. The characterization of the obtained systems was performed by the following techniques: Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), and Superficial Area (BET).

The mesoporous material MCM-41 presented an arrangement hexagonal ordered pores revealed by TEM as shown in the figure 1, the XRD patterns showed four well-resolved reflections characteristic of these systems. The superficial area of this material was 805 m^2/g and pore sizes of 2 nm approximately. Incipient impregnation was carried out with salts of Fe and Co, to obtain the metallic and bimetallic nanoparticles. The XRD patterns for each system indicate the formation of the oxide phases of each metal. For the cobalt the phase formed corresponded to the spinel $CoCo_2O_4$ and for the iron the phase obtained was Fe_2O_3 (hematite). In the case of the bimetallic particles the formation of spinel $FeCo_2O_4$ was observed. Table 1 show the results of superficial area and average particle size for each system. From the superficial area measurements after impregnation it seems that the particles locate in the channels blocking them. Figure 2-a) show the images corresponding to the nanoparticles of FeCo oxide and the figure 2-b) nanoparticles of cobalt oxide, deposited on MCM-41. In these images the formation of small particles and agglomerates can be observed. The particle size for the pure elements was smaller with a narrow size distribution than for the bimetallic FeCo that show a bimodal distribution, however this was due to some particle agglomerates formed outside the molecular sieve. When the particles formed in the channel of the mesoporous material the particle size was very uniform and of 2 nm.

References
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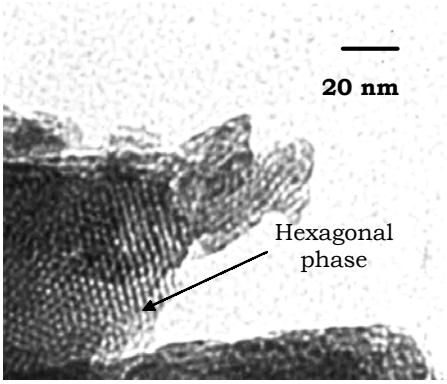
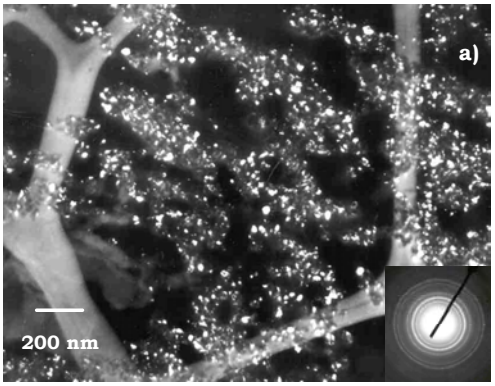


Fig. 1 Bright image of MCM-41



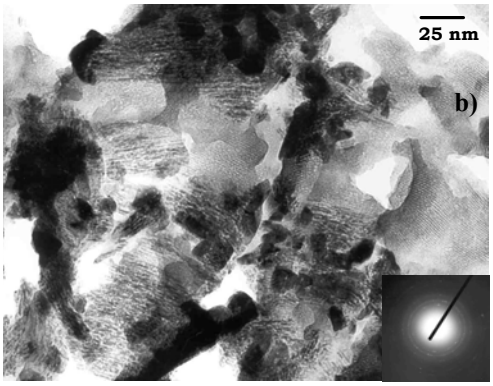


Fig.2 a) Dark image of FeCo b) Bright field image of Co on MCM-41

Table 1

Nanophase Systems	Phase	Average Particle size (nm)	Area BET m^2/g
Fe/MCM-41	Fe_2O_3	3	426
Co/MCM-41	$CoCo_2O_4$	4	540
FeCo/ MCM-41	$FeCo_2O_4$	2 and 20	356