MAGNETIC CHARACTERIZATION OF MANGANESE OXIDE NANOFIBRES

N. Iturriza[1], J.J. del Val[1], J. González[1], C. Gómez-Polo [2], D. Frías[3], P. Navarro[3], M.I.Domínguez[4], J.J. Delgado[5], J.A. Odriozola[4], M. Montes[3]*

[1] Dep. Materials Physics. University of the Basque Country, P. M de Lardizabal, 3 , 20080 San Sebastián, Spain

[1] Dep. Física. Universidad Pública de Navarra, Campus de Arrosadía, 31006 Pamplona, Spain

[13]Dep. de Ingeniería Química. Universidad del País Vasco, P. M de Lardizabal, 3 , 20080 San Sebastián, Spsin

[24]Instituto de Ciencia de Materiales de Sevilla (Centro Mixto US-CSIC), Avda. Américo Vespucio s/n, 41092 Sevilla, Spain

[35]Grupo de Química de Sólidos y Catálisis, Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica y Química, Inorgánica de la Universidad de Cádiz. C/. República Saharaui s/n. Aptdo. 40, Puerto Real. Cádiz. Spain.

Advanced magnetic materials displaying fascinating magnetic properties together with extremely low dimensions are of great interest for technological applications. Consequently, different kinds of magnetic materials have been developed and thoroughly investigated in the last recent years. In this context, manganese oxide compounds that additionally exhibit magnetic behaviour can be considered as very promising candidates to be used in several applications (such as those related with catalysis, ion storage and separation battery electrodes, chemical sensors, and patterning) and, consequently, significant efforts have been expended in a continuous attempt to optimize and improve this kind of materials. Therefore, several characterization techniques have been proposed (microscopy techniques, XRD diffraction and more recently that involve magnetic behaviour).

In this work we deal on the microstructural and magnetic characterization in manganese oxide nanofibres of the OMS (Octahedral Molecular Sieve) family. These materials usually called Cryptomelane were prepared from Mn^{7+} and Mn^{2+} by two methods: reflux and ball milling.

Microstructural characterization was carried out by DRX, N_2 adsorption, HRTEM, XPS and DRITS. KMn_8O_{16} phase is confirmed by XRD in the form of d(110) oriented fibres with diameters from 7 to 15nm and aspect ratio 10-50 presenting high surface area. Mn average oxidation state measured by different techniques varies between 3.66 and 3.94. 2x2 channels of the OMS structure are not accessible because they filled with K and water stabilizing the structure.

Magnetic susceptibility measurements in manganese oxide compounds (Cryptomelane and reference materials, MnO_2 and Mn_2O_3) were performed by means of a SQUID magnetometer. From such measurements, the antiferromagnetic behaviour of the samples was evidenced with Nèel temperature, T_N, below 50 K, while for $T>T_N$, the thermal dependence of the magnetic susceptibility was, satisfactorily, fitted according to

the Curie-Weiss law. The Curie-Weiss law allows estimating the effective magnetic moment, $_{eff}$, of the magnetic Mn atoms and, finally, the atomic fraction of Mn^{3+} and Mn^{4+} in the compound.

The excellent catalytic activity in the total oxidation of acetyl acetate is explained by the non-stoichiometry character (balance between Mn^{3+} and Mn^{4+}) confirmed by different techniques and the textural properties resulting from its nanofibre character.

*) Author responsible