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TITLE: Low Temperature Synthesis, Structure and Magnetism of Manganese Sillenites Nanoparticles

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Digest Body: The research and application of nanomaterials have a significant impact on our development and has been the focus of intense studies due to the promising mesoscopic properties shown by particles of quantum dimensions. One important material family in this scope are the sillenites. The large structural family of bismutates with sillenite structure [1] has technological applications in the fields of electro-optics, acoustics, piezotechniques and as dielectrics in electronic. Stoichiometric sillenites are compounds with the structure $Bi_{12}MO_{20}$, where M is a tetravalent ion. The most studied compounds with this structure have been those with M=Si,Ge and Ti, because of their electro-optical and dielectric properties [2]. Other compounds has also emerged as being of particular interest [3,4]. Sillenites crystallize in the body-centered space group I23, with two formula units per unit cell and the structure consists of deformed Bi-O polyhedra, where Bi ions are coordinated with five oxygen ions, and a MO_4 tetrahedra placed in the center and in the axis of the unit cell.

In this work, a soft chemical route under refluxing conditions [5] has been followed to prepare two polycrystalline manganese sillenite samples, $Bi_{12}MnO_{20}$ (BMO), at a very low temperatures (~ 100 ^{o}C). Sample A was made with Bi_2O_3 in excess, with 6 hours digest, while sample B was made with an equimolar concentration of the precursors, 8 hours of digest.

X-ray diffraction (XRD), obtained on a Bruker D8 Advance X-Ray Diffractometer with CuKa radiation, and Scanning (SEM), obtained on a JEOL JSM-5800LV, show a cubic BMO pure phase with a=10.206 Å, homogeneous morphology and average particle size of 10nm, as indicated in figure 1. The patterns were analyzed using the Rietveld method and they can be indexed to a pure-phase BMO with cubic structure, belonging to the I23 space group, well consistent with the reported data JCP2: 01-082-1024. The sharp diffraction peaks, in both cases, indicates that well-crystallized BMO crystallites could be easily achieved by the present refluxing process. The X-ray line profile, assuming that the broadening of the peaks is determined only by the size of crystallites [6], yields a crystallite size of 40 nm and 27 nm for samples A and B respectively. Using the classical Williamson

Hall method for deconvolution size and strain contributions to line broadening as a function of 2θ, a superior fit was obtained with particle sizes of 130 nm and 58 nm. The strain value for the sample B is slightly higher and can be related to the decrease in the size of crystallites.

The zero-field-cooled (ZFC)/field-cooled (FC) measurements for sample A is shown in figure 2, with a classical nanoparticle behavior, blocking temperature (TB ~ 20K) that varies with the applied dc field and a Curie temperature (T_C) around ~48K. Ac susceptibility measured at different frequencies shows an Arrhenius behavior with and unusual small relaxation time, $\tau_0 \sim 10^{-22}$. This deviation from the expected relaxation time of superparamagnetic nanoparticles ($\tau_0 \sim 10^{-12}$) is recovered when ac susceptibility is measured in a presence of an external dc field and can be attributed to the high surface anisotropy due to the high surface stress, compatible with the X-ray results.

The normalized magnetization versus H/T ratio and the magnetic field dependence of the magnetization does not show a typical superparamagnetic behavior with collapsed M versus H/T curves. Above the blocking temperature, the thermal fluctuation energy (kB) is larger than the uniaxial anisotropy energy (KV). Above T_C the magnetic susceptibility follows a Curie-Weiss law with non-hysteretic features.

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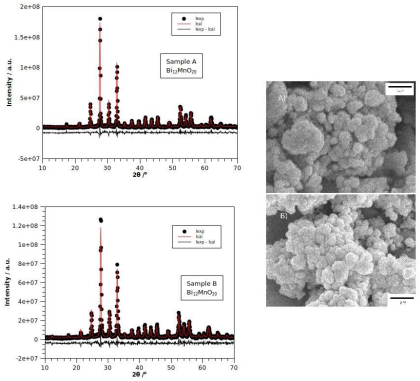
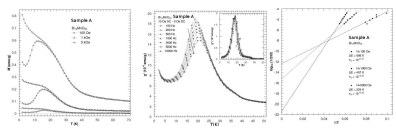


X-ray diffraction pattern, Rietveld analysis and SEM images of the as-prepared samples A and B synthesized by the reffluxing process.



ZFC/FC, ac susceptibility and Arrhenius plot of the as-prepared sillenite

sample A synthesized by the reffluxing process.
