INTERLAYERED MnS NANOCLUSTERS WITHIN A HIGH –CHARGE SYNTHETIC SODIUM MICA (Na-2-MICA)

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INTERLAYERED MnS NANOCLUSTERS WITHIN A HIGH – CHARGE SYNTHETIC SODIUM MICA (Na-2-MICA)

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INTRODUCTION

The synthesis of functionalized nanoscale materials focused on clays as building blocks have been subject of great interest and development in recent decades due to their potential applications in design of heterogeneous[1] catalysts, optical devices, optoelectronic, magnetic and electrochemical[2] applications. The metal nanoclusters are polynuclear aggregates that may contain a few atoms to as many as thousands, whereas they can change their structure around one or more metal atoms; thus, together the manganese-based nanoclusters may display interesting catalytic properties due to the intrinsic variable redox potential of Mn. The growing of metal nanoclusters encapsulated inside porous synthetic aluminosilicates may show significant advantages in comparison with the synthesis starting from natural ones; its high chemical stability and high crystal uniform lattice are outstanding. In this work, a Na-2-mica ($Na_2[Si_6Al_2]Mg_6O_{20}F_4 \cdot xH_2O$) was synthesized by the sol – gel method; it is a swelling layered 2:1 phyllosilicate with both of high layer charge and cation exchange capacity (CEC) 270 meq/100 g of mica.

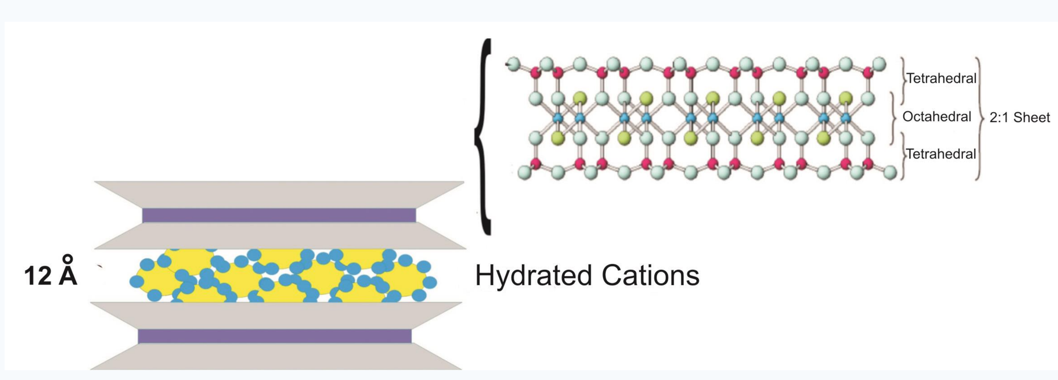
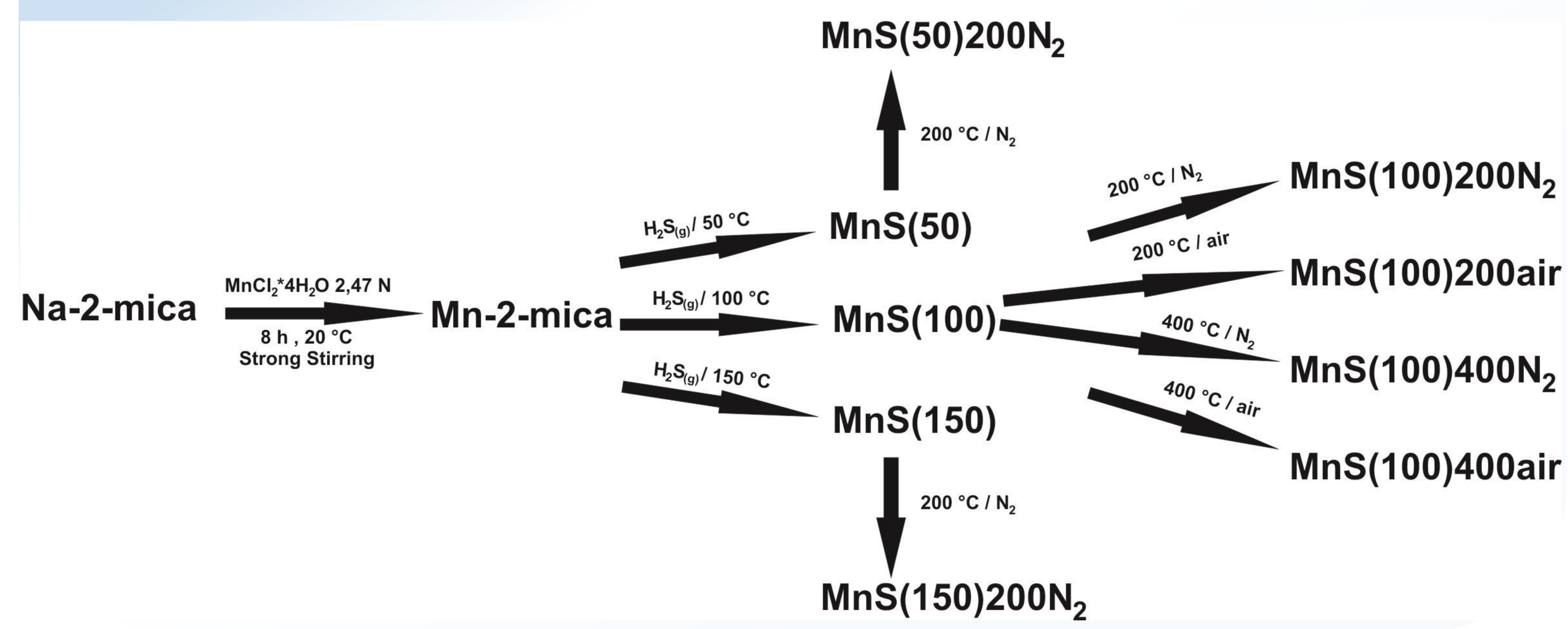


Fig. 1 Na-2-mica

EXPERIMENTAL

Preparation of interlayered MnS nanoclusters: assessment of the effects exerted by sulfidation temperature (50, 100 or 150 °C), post-heating temperature (200 or 400 °C) and type of atmosphere (N_2, air)



RESULTS

Effect of sulfidation temperature on the growing of MnS nanoclusters interlayered in Na-2-mica

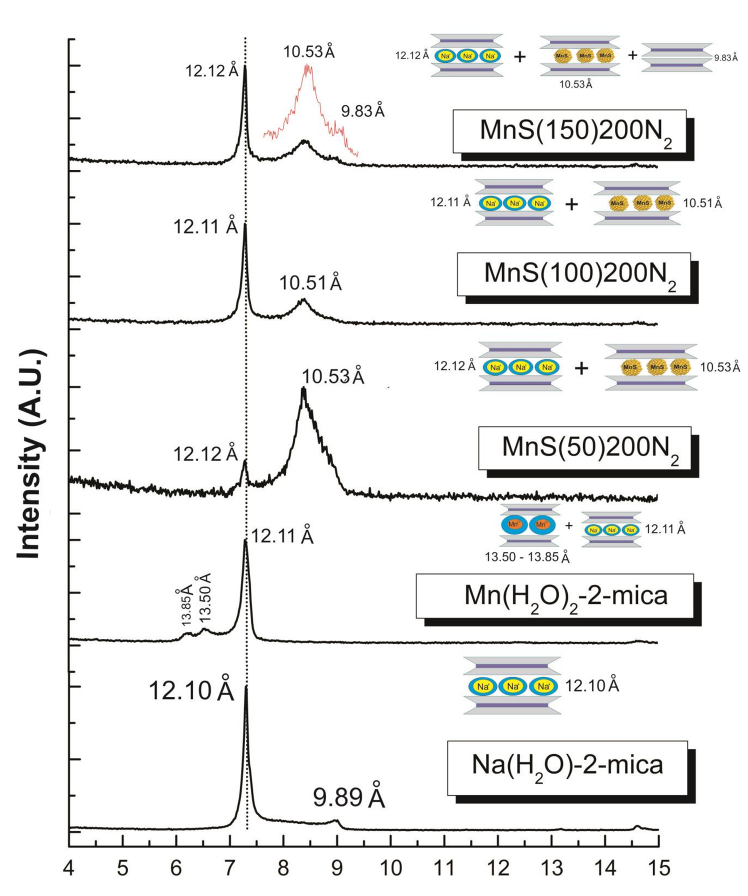


Fig. 2 Powder – XRD: Effect of sulfidation T (50, 100 or 150 ° C) on the basal spacing of the starting Mn-exchanged Na-2-mica.

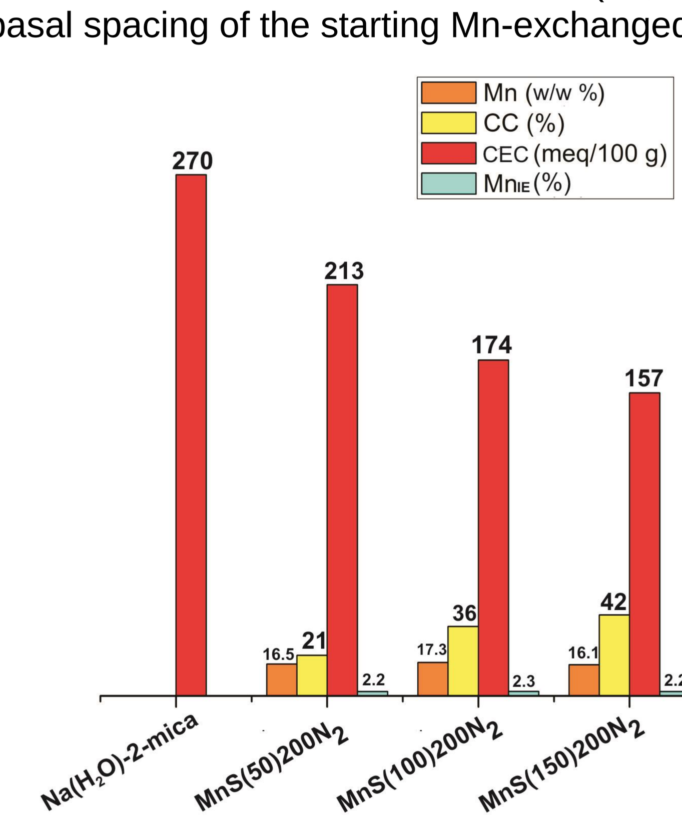


Fig. 3 Cationic exchange capacity (CEC), compensation of starting CEC (CC), Mn content and Mn incorporation efficiency (Mn_{EI}) of sulfidized materials at different temperature (50, 100 or 150 °C).

Effect of type of atmosphere (inert vs. oxidizing) and temperature used in the final thermal treatment on the growing of MnS nanoclusters interlayered in Na-2-mica

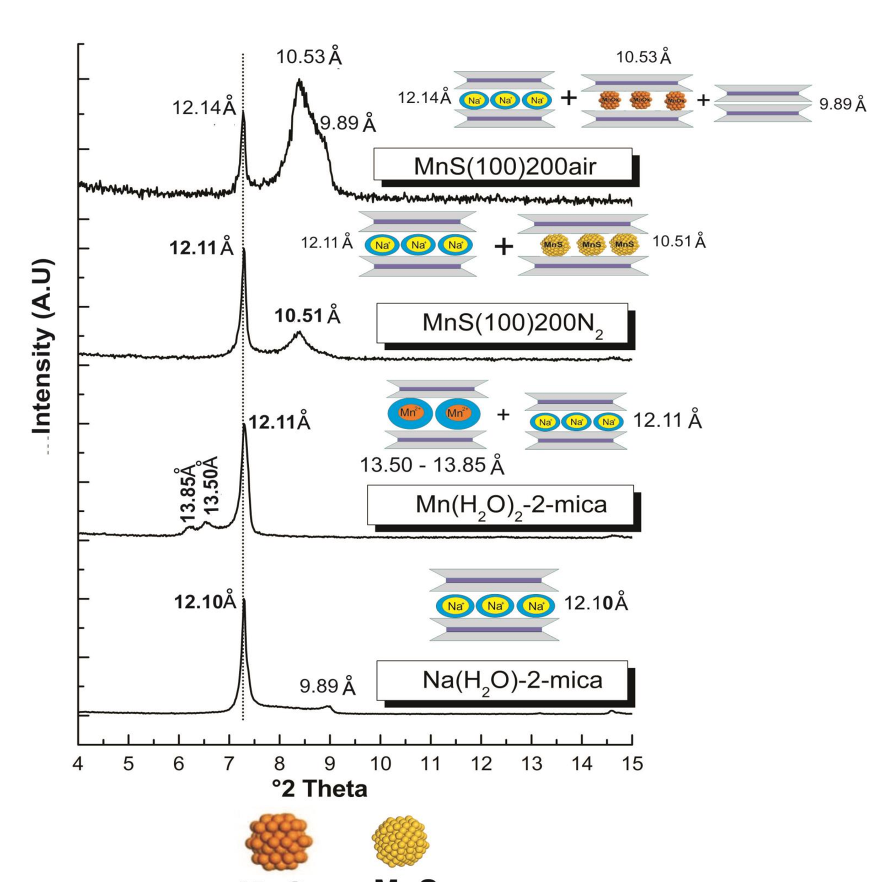


Fig. 4 Powder – XRD: Effect of the atmosphere (N_2 vs. air) in the final thermal treatment (T = 200 °C) on the basal spacing of the starting Mn-exchanged Na-2-mica.

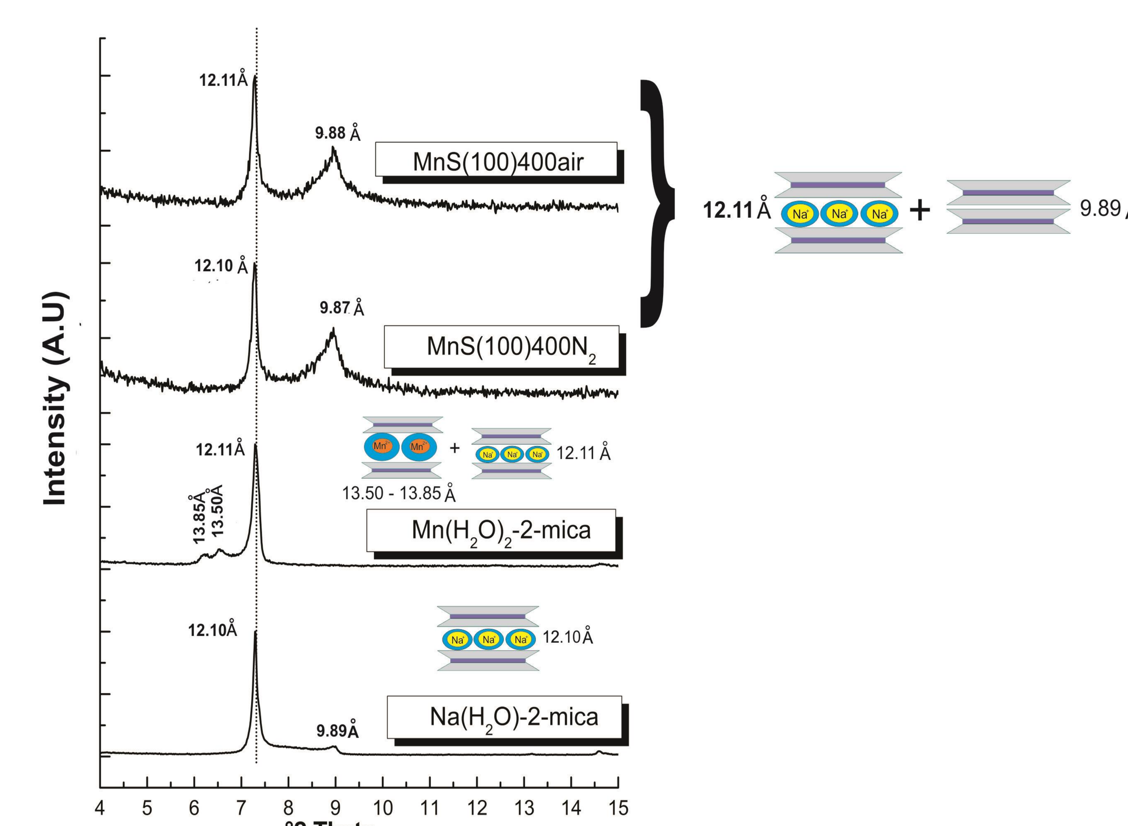


Fig. 5 Powder – XRD: Effect of the atmosphere (N_2 vs. air) in the final thermal treatment (T = 400 °C) on the basal spacing of the starting Mn-exchanged Na-2-mica.

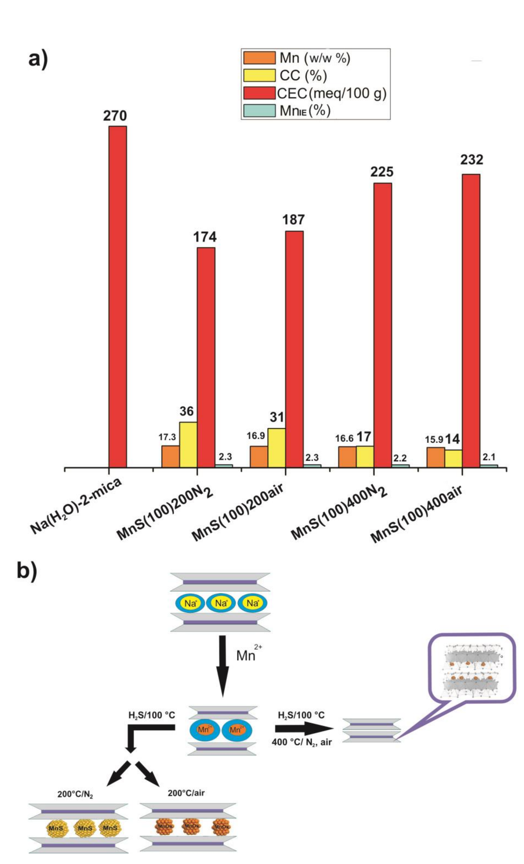


Fig. 6 a) Cationic exchange capacity (CEC), compensation of the starting CEC (CC), Mn content and Mn incorporation efficiency (Mn_{EI}) of sulfidizated materials heated under different temperatures and atmospheres; b) Sketch summarizing main results in preparation of MnS nanoclusters as a function of temperature and atmosphere of final heating treatment.

CONCLUSION

The development of nanoclusters of MnS confined into the interlayer space of *Na-2-mica* was found to be favored at low sulfidation temperatures, since high temperatures at this step produce collapse of the 2:1 layers. As the final heating is performed under oxidizing atmosphere, MnO_x aggregates are formed having similar dimensions as former MnS. Such aggregates affect the compensation of the starting CEC as well as the ability to stabilize the metal; therefore, in this sense the most appropriate atmosphere for the generation and stabilization of the MnS nanoclusters is nitrogen. The heating process after sulfidation suggested that irrespective the nature of the atmosphere used (inert or oxidizing), MnS or MnO_x particles were formed apparently in a microencapsulated conformation. Furthermore, formation of aggregates was favored at low calcination temperatures (200 °C), because higher temperatures (400 °C) independently of the atmosphere used promoted development of the anhydrous phase of the mica, that is, material's collapse.

Acknowledgment

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