In-situ growing of MnS and FeS nanoclusters at the interlayer of Al-pillared bentonite

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Introduction

Since formation of metal nanoclusters is thermodynamically unstable and difficult to control, in this work it has been explored the *in-situ* growing of either MnS or FeS nanoclusters in the interlayer space of a bentonite by means of a pretty short process taking only around 12 h. The interlayered polynuclear sulfidized metal clusters were prepared by cationic exchange of either Mn^{2+} or Fe^{2+} on the bentonite previously interlayered/pillared with aluminium under different conditions. These metal sulfidized nanomaterials have attracted substantial interests due to their unique optical and electrical properties and wide variety of potential applications in electroluminescence[1] and nonlinear optical devices[2]. Since the main physical and optical properties of such metal sulfides primarily depend on their shape and size, the immobilization of metal sulfide nanoparticles in a spatially confined environment is a way to control the photo-physical and photo-chemical properties which result in very interesting strategy of morphological control.

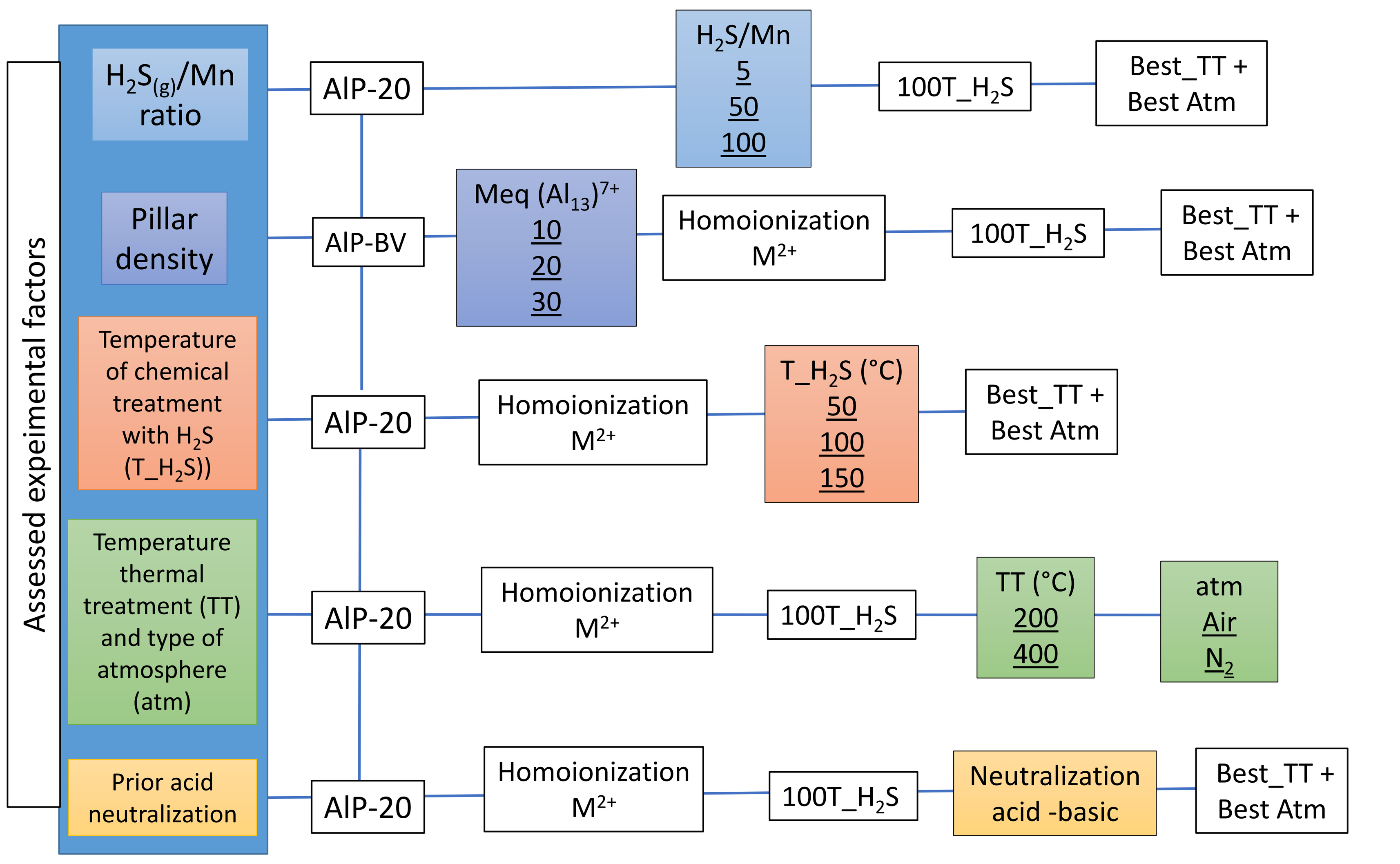

Figure 1. Sketch of prepared materials.

Experimental Materials & Methods

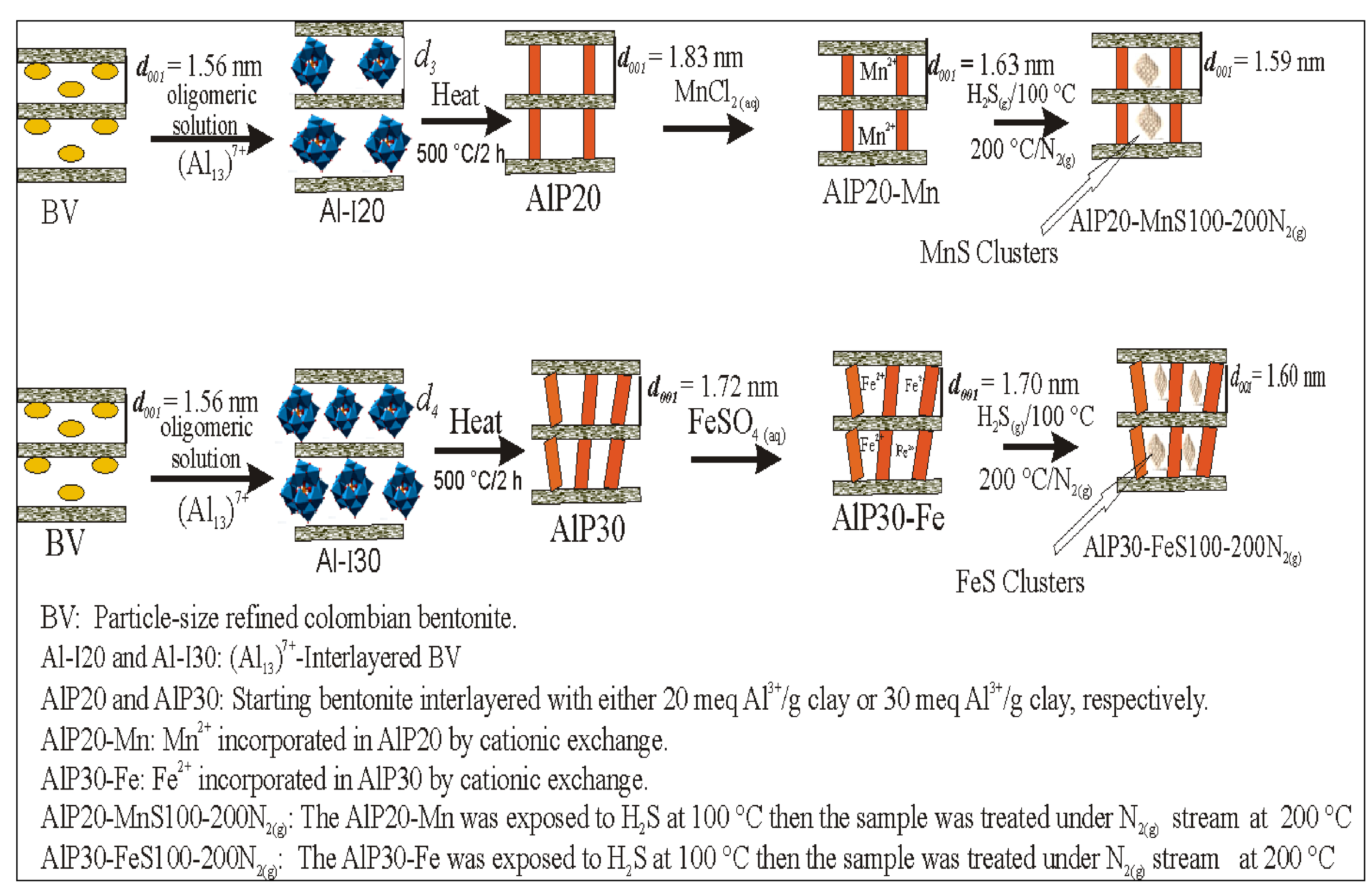

Figure 2. Preparation of materials by in situ growing of either MnS or FeS interlayered nanoclusters.

Results

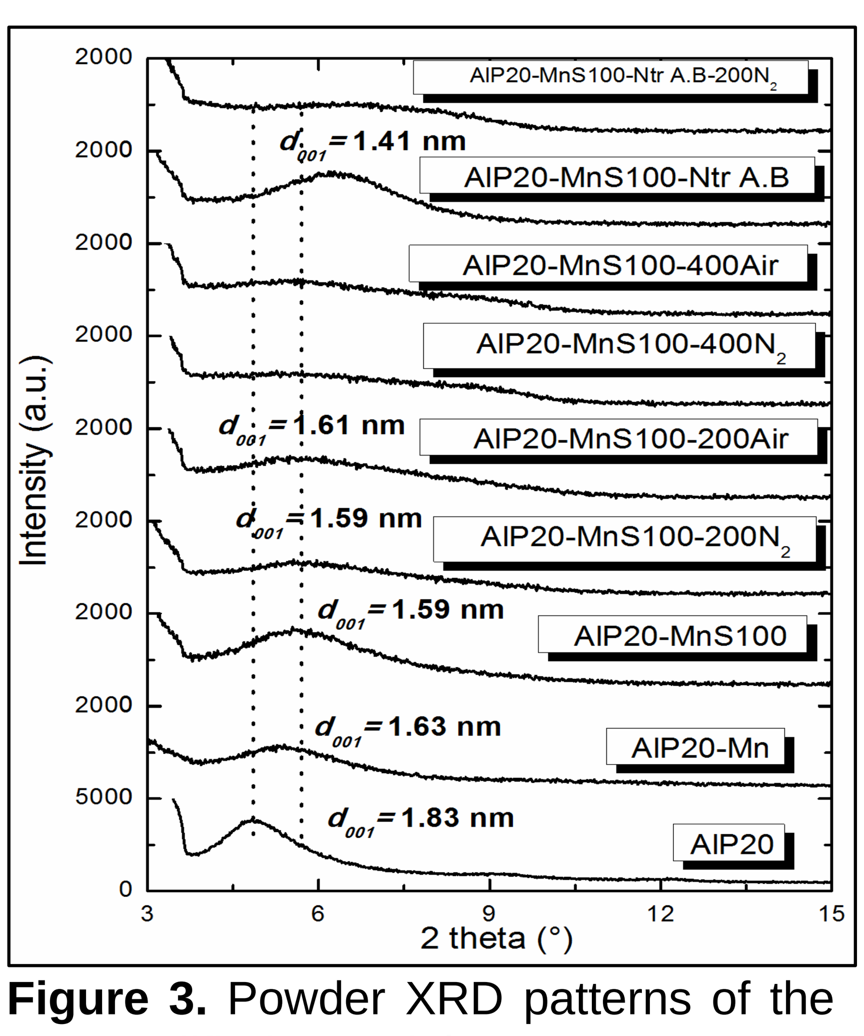

Figure 3. Powder XRD patterns of the Mn–modified materials.

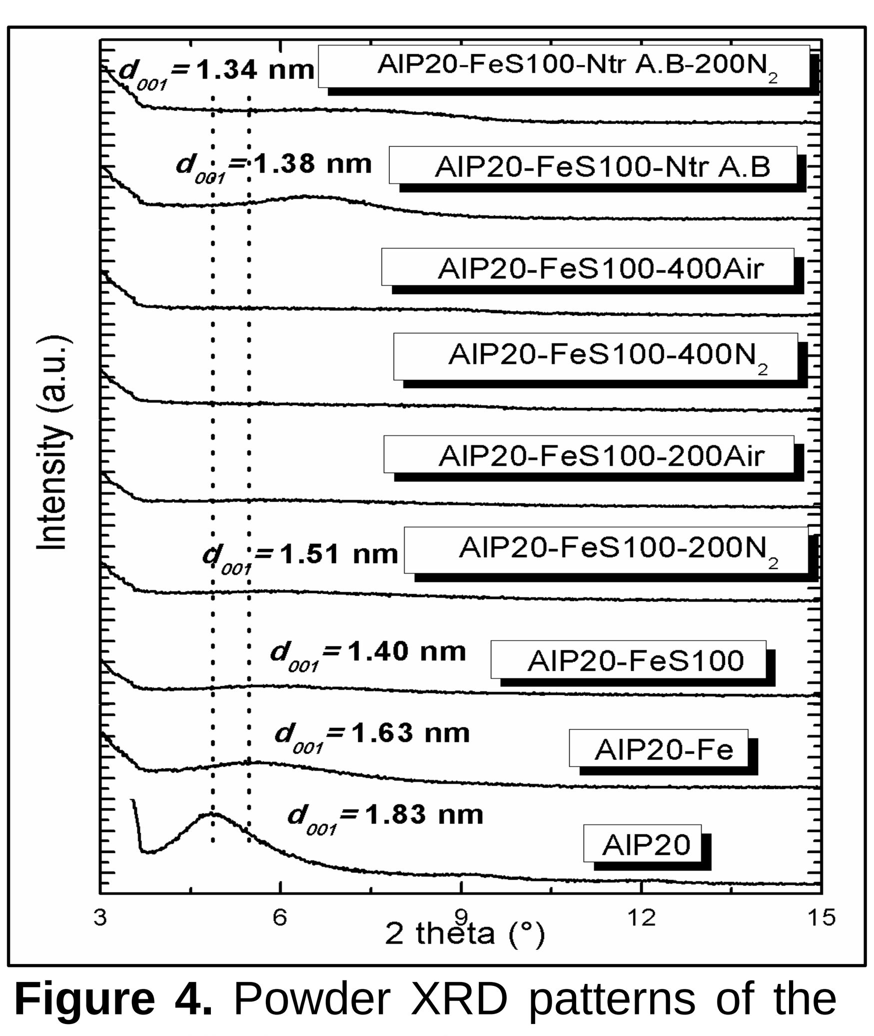

Figure 4. Powder XRD patterns of the Fe–modified materials.

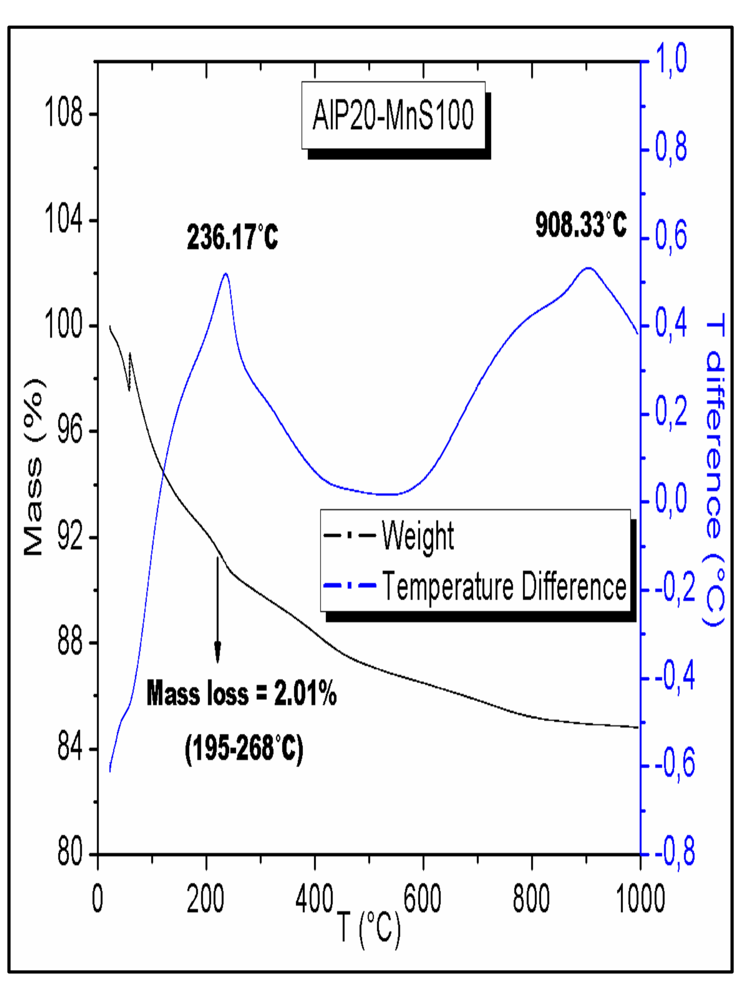

Figure 5. Thermal analysis diagram (TGA/DSC) AlP20-MnS100

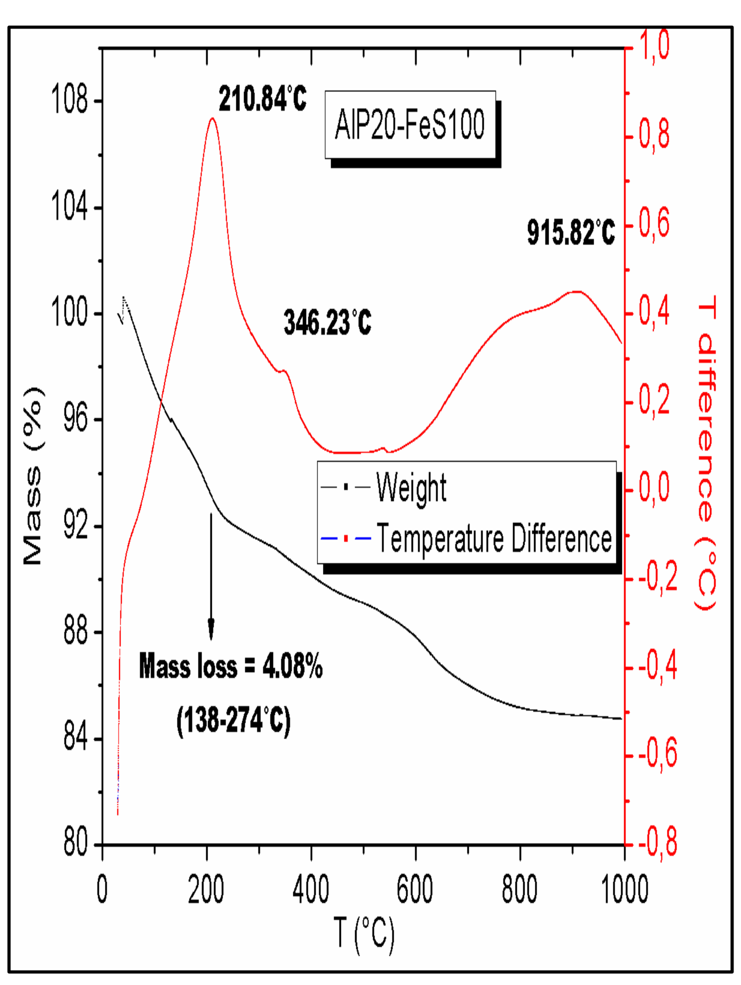

Figure 6. Thermal analysis diagram (TGA/DSC) AlP20-FeS100

Table 1. SiO_2-normalized content of Mn, CEC, compensation of cationic exchange capacity and textural properties of the materials.

Sample	Content (wt.%)	CEC (meq/100 g)	S_{BET} (m^2/g)
	MnO_2/SiO_2		
AlP20	0.00	34	107
AlP20-Mn	0.13	45	………
AlP20-MnS50-200N_2	0.07	11	84
AlP20-MnS100-200N_2	0.05	8	85
AlP20-MnS150-200N_2	0.07	10	33
AlP20-MnS100-400N_2	0.05	14	………

Table 2. SiO_2-normalized content of Fe, CEC, compensation of cationic exchange capacity and textural properties of the materials.

Sample	Content (wt.%)	CEC (meq/100 g)	S_{BET} (m^2/g)
	Fe_2O_3/SiO_2		
AlP20	0.04	34	107
AlP20-Fe	0.09	36	………
AlP20-FeS50-200N_2	0.08	2	26
AlP20-FeS100-200N_2	0.12	2	………
AlP20-FeS150-200N_2	0.08	6	………
AlP20-FeS100-400N_2	0.11	6	………
AlP30-Fe	0.11	7	………
AlP30-FeS100-200N_2	0.07	6	………

Conclusions

- The most suitable conditions for the *in-situ* growing of MnS nanoclusters interlayered in Al-pillared bentonite were established: molar ratio $(H_2S_{(g)})$/Mn (interlayered) = 50; T of chemical treatment = 100 °C; T of thermal treatment = 200 °C.
- The most appropriate conditions for the growth of interlayered FeS nanoclusters in Al-pillared bentonite were: molar ratio $(H_2S_{(g)})$/Fe (interlayered) = 50; aluminum content = 20 mequiv. Al^{3+} g^{-1}; T of chemical treatment = 100 °C; T thermal treatment = 200 °C.
- The type of atmosphere, either oxidizing or inert, did not display significant effect on the structural properties of the resulting materials.

Acknowledgement

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References

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[2] Chin, A.H., Calderón, O.G., Kono, J., 2001. Phys. Rev. Lett. 86, 3292–3295.