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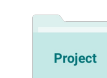


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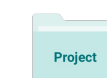
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# In-situ growing of MnS and FeS nanoclusters at the interlayer of Al-pillared bentonite



H.J. Muñoz<sup>a\*</sup>, A.M. García<sup>a,b</sup>, A. Gil<sup>b</sup>, M.A. Vicente<sup>c</sup> and L.A. Galeano<sup>a\*</sup>

Corresponding author : \*alejandrogaleano@udenar.edu.co



<sup>a</sup> Research Group on Functional Materials and Catalysis. Department of Chemistry, University of Nariño, Calle 18, Cra 50 Campus Torobajo, Pasto Colombia.

<sup>b</sup> Department of Applied Chemistry, Public University of Navarra, Edificio Los Acebos, Campus Arrosadía, 31006-Pamplona, Spain.

<sup>c</sup> Department of Inorganic Chemistry, Chemical Science Faculty, Salamanca University, P. de la Merced, s/n, 37008 - Salamanca, Spain.

## 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<sup>2+</sup> or Fe<sup>2+</sup> 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<sup>1</sup> and nonlinear optical devices<sup>2</sup>. 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.

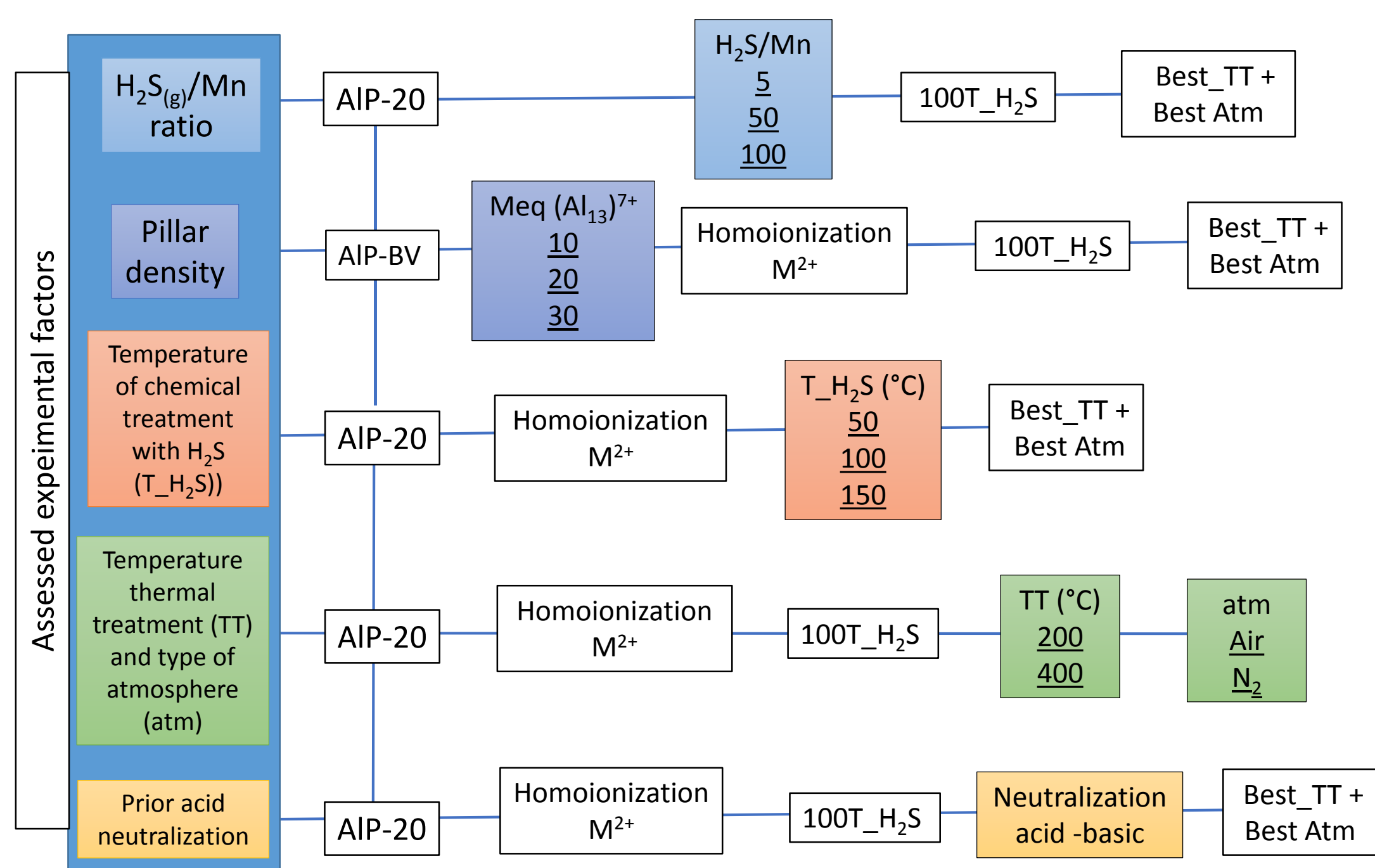
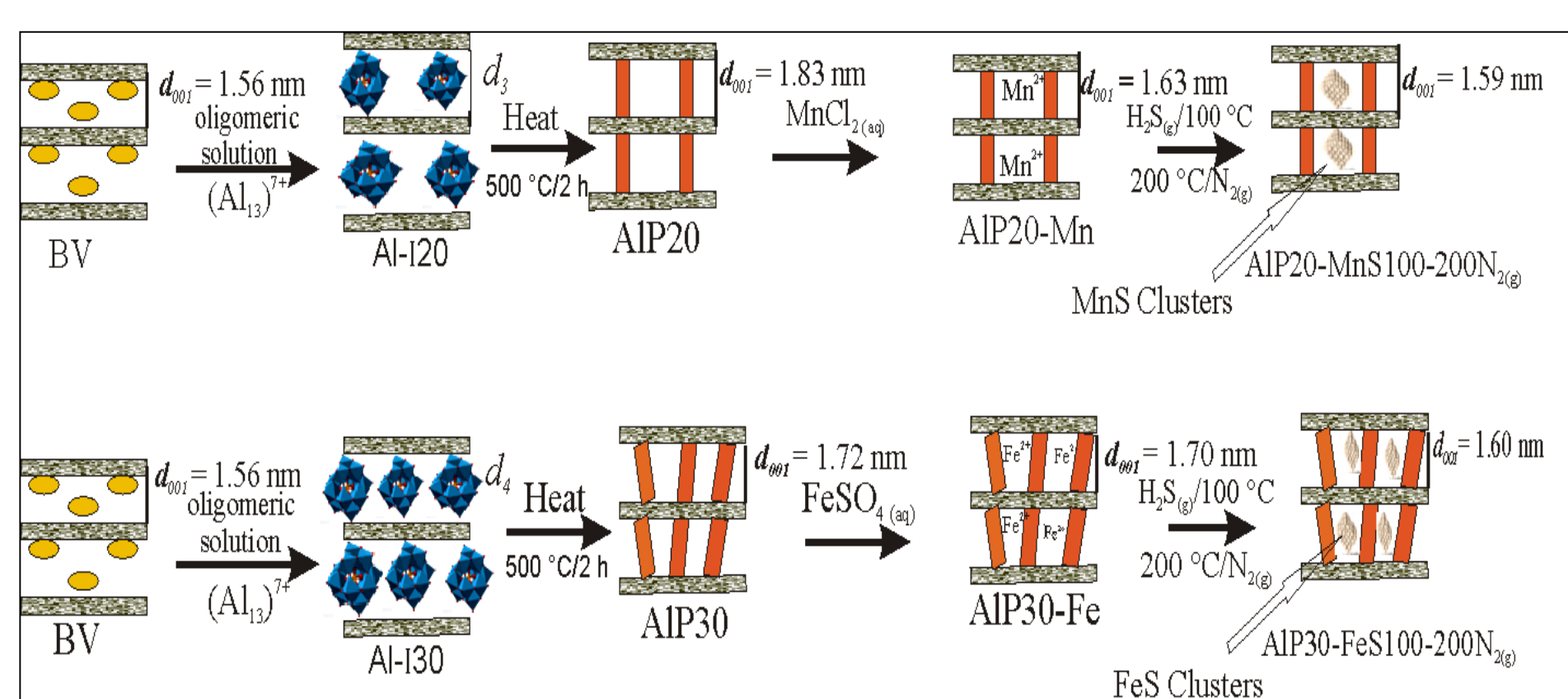


Figure 1. Sketch of prepared materials.

## Experimental Materials & Methods



BV: Particle-size refined colombian bentonite.  
 Al-120 and Al-130: (Al<sub>13</sub>)<sup>7+</sup>-interlayered BV  
 AIP20 and AIP30: Starting bentonite interlayered with either 20 meq Al<sup>3+</sup>/g clay or 30 meq Al<sup>3+</sup>/g clay, respectively.  
 AIP20-Mn: Mn<sup>2+</sup> incorporated in AIP20 by cationic exchange.  
 AIP30-Fe: Fe<sup>2+</sup> incorporated in AIP30 by cationic exchange.  
 AIP20-MnS100-200N<sub>2</sub>: The AIP20-Mn was exposed to H<sub>2</sub>S at 100 °C then the sample was treated under N<sub>2</sub> stream at 200 °C  
 AIP30-FeS100-200N<sub>2</sub>: The AIP30-Fe was exposed to H<sub>2</sub>S at 100 °C then the sample was treated under N<sub>2</sub> stream at 200 °C

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

## Results

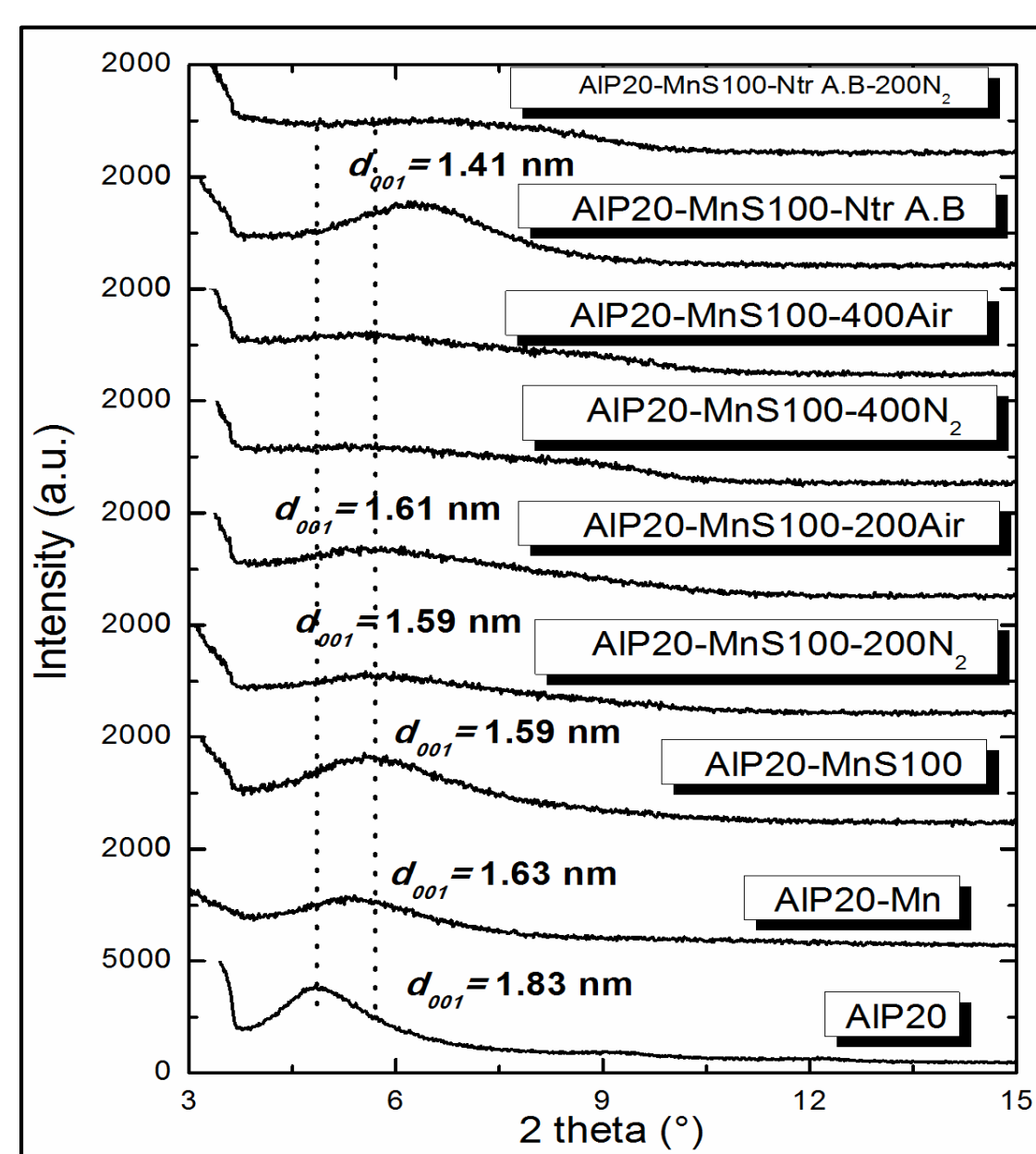


Figure 3. Powder XRD patterns of the Mn-modified materials.

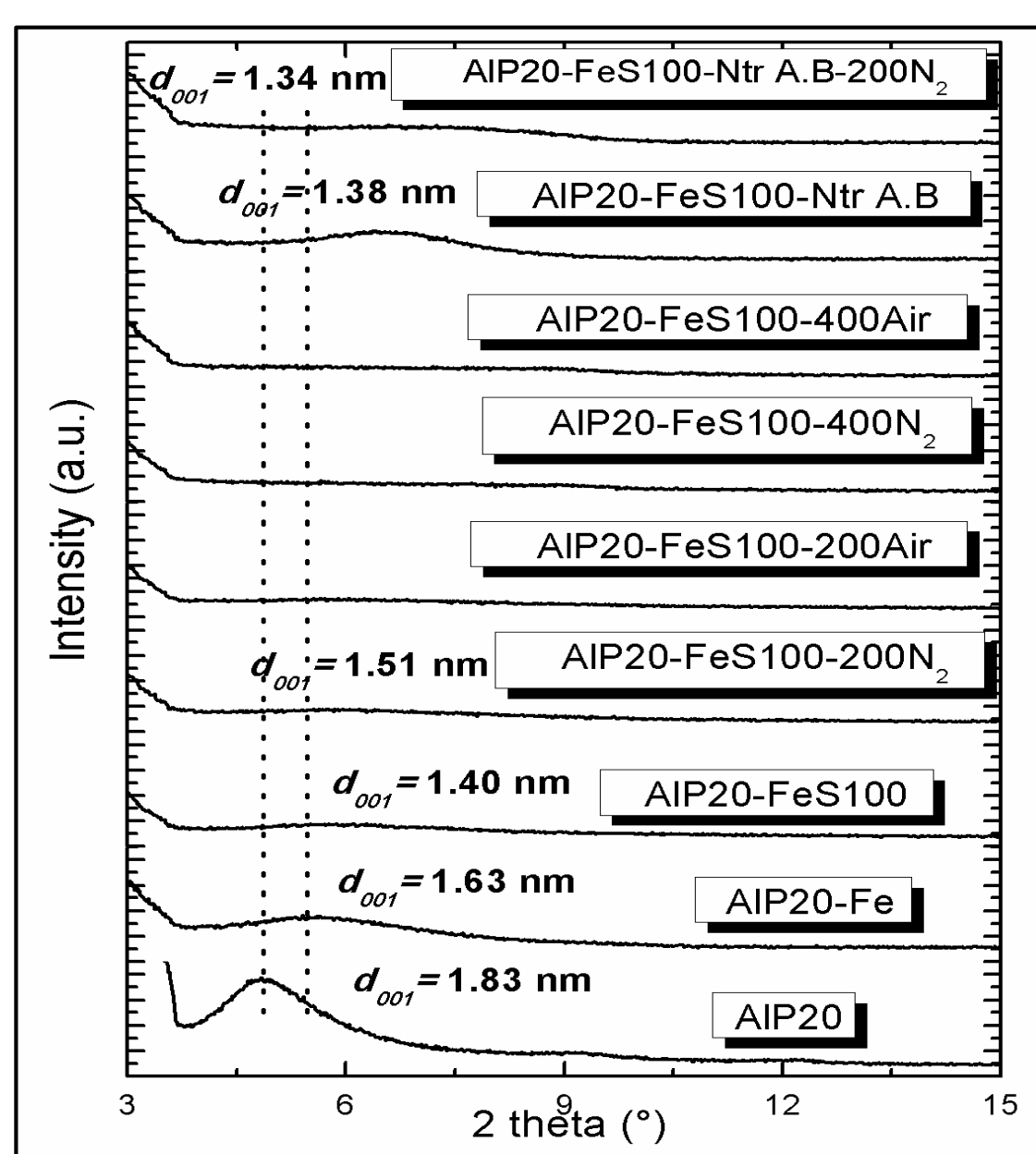


Figure 4. Powder XRD patterns of the Fe-modified materials.

Table 1. SiO<sub>2</sub>-normalized content of Mn, CEC, compensation of cationic exchange capacity and textural properties of the materials.

| Sample                         | Content (wt.%)                     | CEC (meq/100 g) | S <sub>BET</sub> (m <sup>2</sup> /g) |
|--------------------------------|------------------------------------|-----------------|--------------------------------------|
|                                | MnO <sub>2</sub> /SiO <sub>2</sub> |                 |                                      |
| AIP20                          | 0.00                               | 34              | 107                                  |
| AIP20-Mn                       | 0.13                               | 45              | .....                                |
| AIP20-MnS50-200N <sub>2</sub>  | 0.07                               | 11              | 84                                   |
| AIP20-MnS100-200N <sub>2</sub> | 0.05                               | 8               | 85                                   |
| AIP20-MnS150-200N <sub>2</sub> | 0.07                               | 10              | 33                                   |
| AIP20-MnS100-400N <sub>2</sub> | 0.05                               | 14              | .....                                |

Table 2. SiO<sub>2</sub>-normalized content of Fe, CEC, compensation of cationic exchange capacity and textural properties of the materials.

| Sample                         | Content (wt.%)                                   | CEC (meq/100 g) | S <sub>BET</sub> (m <sup>2</sup> /g) |
|--------------------------------|--|-----------------|--------------------------------------|
|                                | Fe <sub>2</sub> O <sub>3</sub> /SiO <sub>2</sub> |                 |                                      |
| AIP20                          | 0.04   | 34              | 107                                  |
| AIP20-Fe                       | 0.09   | 36              | .....                                |
| AIP20-FeS50-200N <sub>2</sub>  | 0.08   | 2               | 26                                   |
| AIP20-FeS100-200N <sub>2</sub> | 0.12   | 2               | .....                                |
| AIP20-FeS150-200N <sub>2</sub> | 0.08   | 6               | .....                                |
| AIP20-FeS100-400N <sub>2</sub> | 0.11   | 6               | .....                                |
| AIP30-Fe                       | 0.11   | 7               | .....                                |
| AIP30-FeS100-200N <sub>2</sub> | 0.07   | 6               | .....                                |

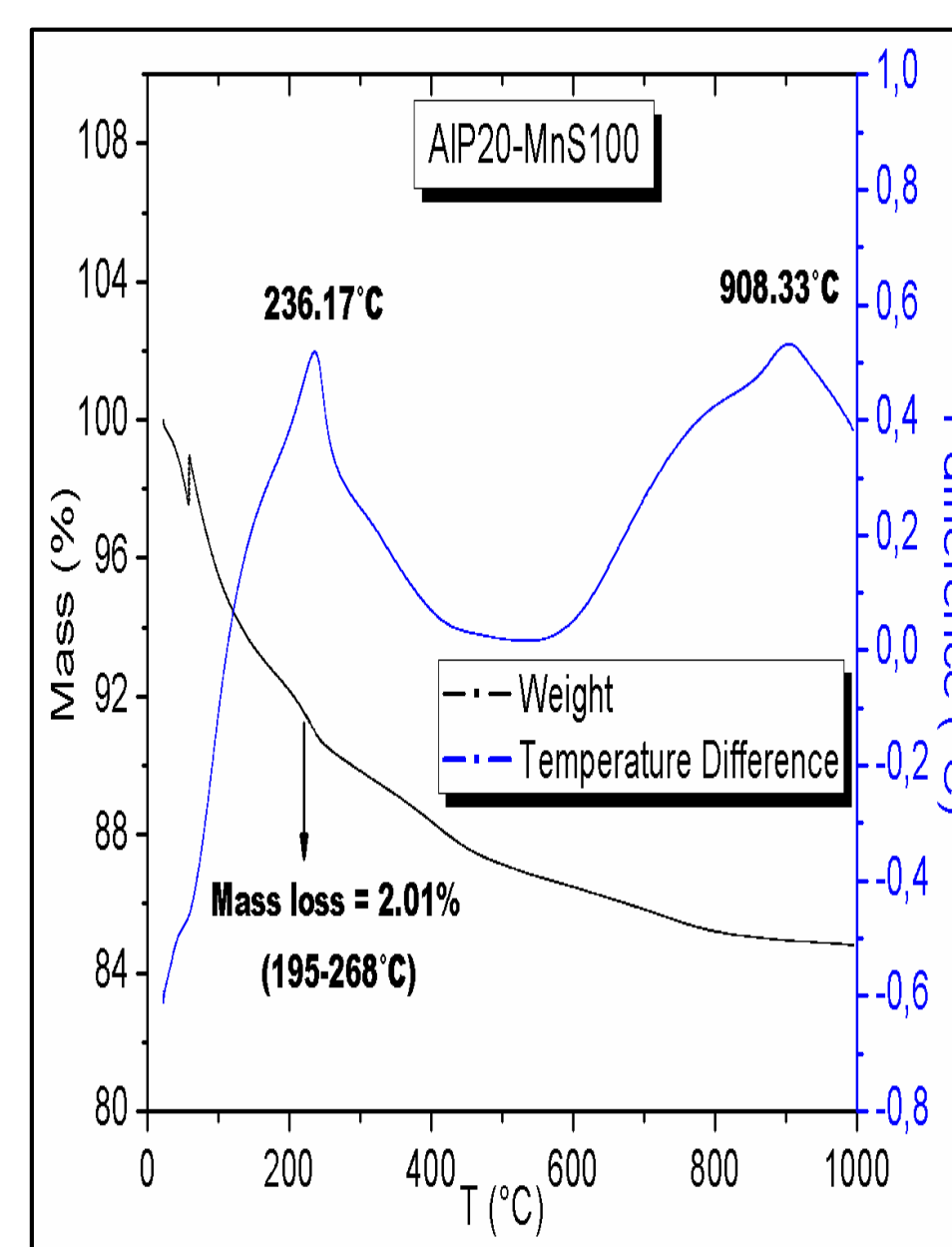


Figure 5. Thermal analysis diagram (TGA/DSC) AIP20-MnS100

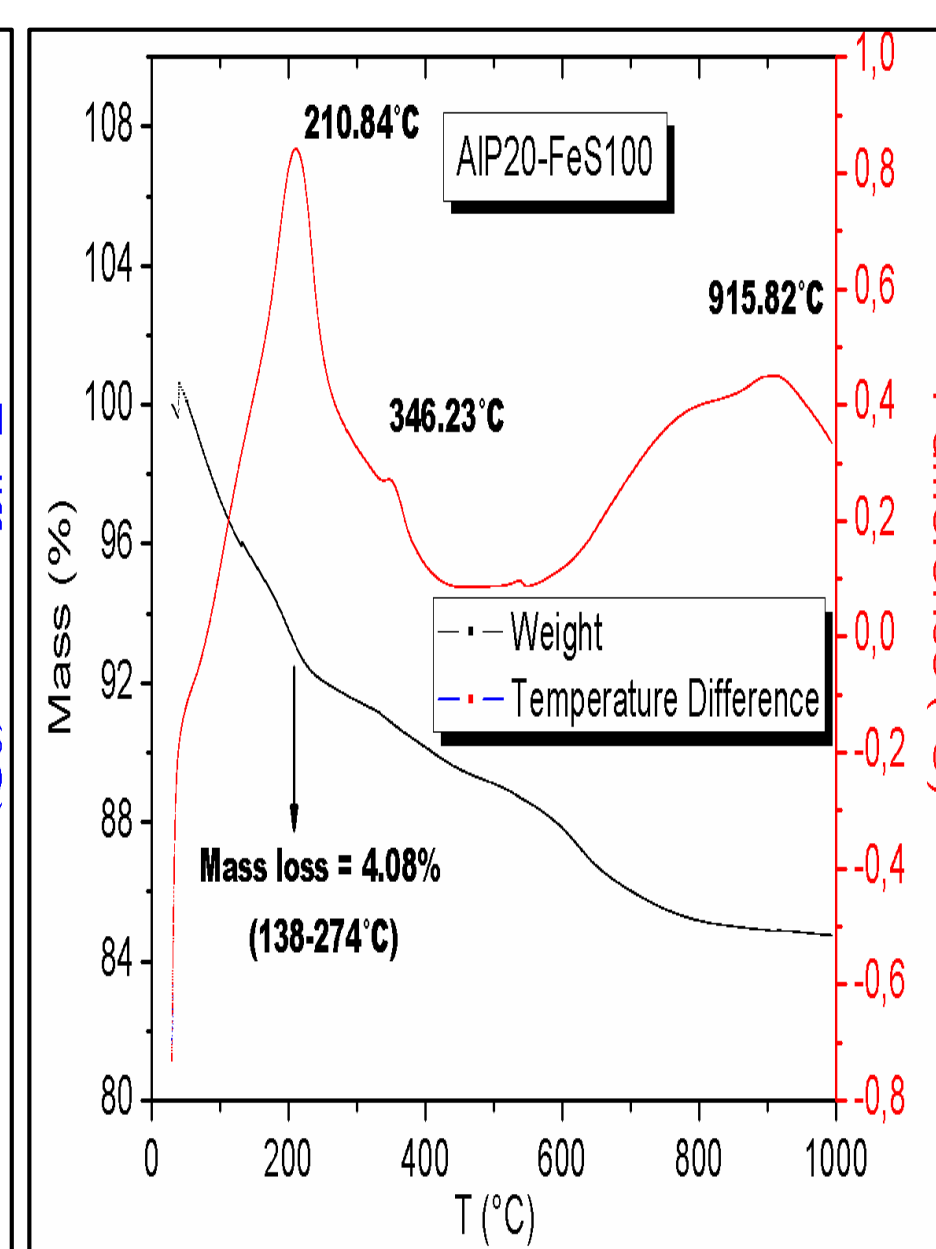


Figure 6. Thermal analysis diagram (TGA/DSC) AIP20-FeS100

## Conclusions

- ❖ The most suitable conditions for the *in-situ* growing of MnS nanoclusters interlayered in Al-pillared bentonite were established: molar ratio (H<sub>2</sub>S<sub>(g)</sub>)/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<sub>2</sub>S<sub>(g)</sub>)/Fe (interlayered) = 50; aluminum content = 20 mequiv. Al<sup>3+</sup> g<sup>-1</sup>; 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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