

## Preparing the experimental report

The experimental report must present the data obtained for each type of nanomaterial synthesized and comment on the different information you can get from complementary characterizations. At the beginning of each paragraph, present in summary the procedure adopted for the synthesis of the materials, pointing the attention on the experimental evidence observed during the procedures. Information from available literature can be used to comment on the results of the characterization techniques employed.

Note: All the XRD patterns uploaded in Moodle2 contain the contribution of  $K\alpha_1$  and  $K\alpha_2$ . Do not forget to remove  $K\alpha_2$  before fitting the patterns.

### **Analysis of Ti-MCM-41**

Perform analysis of physisorption data for each sample. Present in details the analysis performed on your sample (all graphs and comments accordingly to the Procedure for physisorption data analysis) and summarize in a Table the results for all the samples.

Plot the XRD data in the low angle range and in the wide angle range (present the graphs of overlapped patterns). Calculate the (100) distance from the maximum of reflection at low angle (if present). Comment on the crystallinity and phase composition of the materials from data in the wide angle range.

In a final paragraph, present the general comments on this type of nanomaterials, pointing attentions on the following aspects:

1. Why the Ti-MCM-41 samples can be considered within the nanomaterials?
2. Which is the effect of swelling agents on the texture of the Ti-MCM-41? Suggest why.

### **Analysis of TiO<sub>2</sub> samples**

Perform analysis of physisorption data for each sample. Present in details the analysis performed on your sample (all graphs and comments accordingly to the Procedure for physisorption data analysis) and summarize in a Table the results for all the samples.

Plot the XRD data in the wide angle range (present the graphs of overlapped patterns). Perform qualitative analysis with phase identification. Perform Rietveld analysis for quantitative determination of the phase composition for the 3 samples. At end of Rietveld analysis, export the profile of each phase, the phase composition (as %vol) and the cell parameters.

Convert the composition from vol% to wt% (the densities of the TiO<sub>2</sub> polymorphs will be required).

Use the profile of each phase from Rietveld analysis for the calculation of average crystallite size using the Scherrer's equation.

Summarize the results in a table, presenting the results from physisorption analysis (Surface area, pore size and cumulative pore volume) and from XRD analysis (phase composition at wt%, cell parameters in comparison with the values from tabulated reference materials, crystallite size of the different phases).

In a final paragraph, present the general comments on this type of nanomaterials, pointing attentions on the following aspects:

1. Why the TiO<sub>2</sub> samples can be considered within the nanomaterials?
2. Which is the effect of the HNO<sub>3</sub>/Ti molar ratio on the properties of TiO<sub>2</sub> samples? Which are the properties that are majorly affected? Suggest why, taking into account the mechanism of sol-gel processes.

### **Analysis of thiol-protected Au NPs**

The size of thiol-protected Au NPs can be determined by various techniques.

Present the DLS profiles obtained for the materials reporting the average hydrodynamic diameter and its standard deviation.

Present the <sup>1</sup>H-NMR spectra and the calculation of core size from peak broadening.

Present the calculation of the mean crystallite size from the Au (111) reflection of XRD patterns.

Summarize, in a table, the obtained results for the size determination obtained from the different techniques and for the different thiol/Au ratios.

In a final paragraph, present the general comments on this type of nanomaterials, pointing attentions on the following aspects:

1. Which is the effect of thiol/Au molar ratio on the size of Au NPs?
2. Why different techniques give different results? Comment and justify the results.

### **Analysis of Mn-doped ZnS samples**

Present the DLS profiles obtained for the materials reporting the average hydrodynamic diameter and its standard deviation. Comment the differences, if any, between the expected data and the one obtained considering the appearance of the QD solution/suspension.

Analyze the fluorescence data to demonstrate the ability of Mn-doped ZnS QDs as sensor for Co<sup>2+</sup> according to specific instructions.

### **Analysis of commercial silica nanoparticles**

Present the DLS profiles obtained for the silica nanoparticles reporting the average hydrodynamic diameter and its standard deviation. Comment the differences in the observed diameter with the dilution of the sample.