

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 and 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 and MCM-41 samples can be considered within the nanomaterials?
2. Which is the effect of swelling agents on the texture of the samples? Suggest why.
3. Comment on the effect of the presence of Ti on the textural properties of the samples.

Analysis of TiO₂ 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 TiO₂ 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_2 samples can be considered within the nanomaterials?
2. Which is the effect of the HNO_3/Ti molar ratio on the properties of TiO_2 samples? Which are the properties that are majorly affected? Suggest why, taking into account the mechanism of sol-gel processes.
3. A few synthetic conditions have been repeated by 2 students. Comment on reproducibility of synthetic procedures.

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 ^1H -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 CdS 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.

Calculate the average crystallite size of CdS QDs by broadening of main XRD reflection. CdS QDs can be considered to have a cubic crystal structure: as a first approximation, fit the broadening of the main reflection as a single peak.

N.B. Only 3 samples are available for XRD

Calculate the average CdS QDs size by the position of maximum of emission spectra. Refer to the slides used during the presentation of synthetic procedures for the data analysis formula.

Compare the data obtained from the three analysis techniques, summarizing the result in a table.

1. Why different techniques give different results? Comment and justify the results.
2. Is it possible to evidence a correlation between the QDs sizes and the temperature used for synthesis? Explain your answer.

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.