PRELIMINARY STUDIES ON Pb ADSORPTION BY NANO-CRYSTALLINE MAGHEMITE AND HEMATITE STRUCTURES

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Iron oxides, mainly hematite and maghemite, have been known to have promising adsorption capacities towards heavy metals, especially when in the nano-sized-region. This feature brought interest for potential applications in water remediation and environmental conservation purpose. Despite this, there is a lack of information about the fate of the adsorbed metals once they are inside the crystalline structure. For instance, this study works in this optic, trying to set the bases for a better understanding of the adsorption (and retention) process.

The material was synthetised by sol-gel method and secondly analysed with X-ray powder diffraction, showing the presence of both hematite (38.4%) and maghemite (61.6%). The powder, then, was used for the adsorption of lead with different concentrations (from 500 mg/l up to 10000 mg/l), thus confirming the excellent adsorption properties. A mild thermal treatment (150-200 °C) helped in the cation incorporation within the structure. Lead cations indeed enter the structure (especially of maghemite, much less of hematite), as shown in figure 1, where EDS analysis of maghemite nanocrystals display the presence of lead. Moreover, Rietveld refinements of the powder diffraction patterns show an enlarged unit cell (for both maghemite and hematite). The inclusion of lead in the structure is also demonstrated by the retention properties of the material (after contact with an acidic solution with a pH of 5.5), which are very close to 100% (and always larger than 98%).

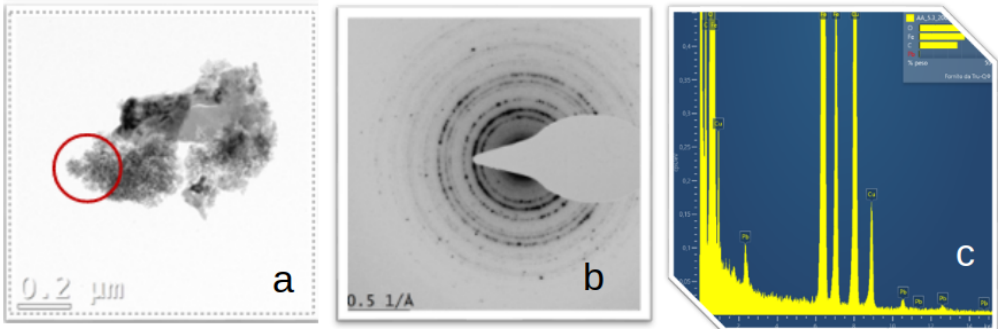

Figure 1 – (a) Image of a cluster of maghemite (small grain size) and hematite (larger grain on the right-hand side); (b) diffraction of the area within the red circle; (c) EDS analysis of the area within the red circle

The powders, after the adsorption process, were analysed at the beamline ID22 (@ESRF) to get total scattering data. The corresponding Pair Distribution Functions show a change in the distances corresponding to the first three coordination shells (a larger change was observed for maghemite with respect to hematite), in agreement with the different amount of lead adsorbed by the two oxides. The reasons for the enhanced performances of maghemite can also be found in its small grain size, with consequent larger surface disorder. It is clear, from the preliminary analysis of the total scattering data, that there is a certain amount of distortion in the structure, ultimately due to the presence of lead cations. What is still lacking (and what needs to be study in much better detail) is a quantitative structural evaluation of the presence of lead. This will be the target of the future investigations, implemented with analyses under different pH and T conditions.