OBTAINING OF $FeCO_3$ MICROPARTICLES

Marius Chirita[1], Mihaela Luminita Kiss[1], Adrian Ieta[2]

[1]*Department of Nanocrystal Synthesis, National Institute for Research and Development in Electrochemistry and Condensed Matter, Timisoara, Plautius Andronescu Str. No. 1, RO-300224, Timisoara, Romania; tel. 0040256494413*
[2]*Department of Physics, State University of New York at Oswego, Oswego, NY*
e-mail: chirifiz@yahoo.com

Abstract
Usinghydrothermal decomposition of the Fe(III)-EDTA complex in the presence of urea, we developed a new procedure for synthesizing highly crystalline $FeCO_3$ starting from Ferric Ammonium Sulfate and Na_4EDTA as main precursors. Single phase $FeCO_3$ microcrystals with size in the range of 50µm-200µm have been obtained after high pressure-temperature treatment time between 15 hours and 26 hours at 230ºC and 250ºC.

Keywords: $FeCO_3$, iron carbonate, hydrothermal decomposition, Fe-EDTA complex.

Introduction
Due to its implications upon the geological sequestration of CO_2[1], the thermodynamics of iron carbonate ($FeCO_3$) has been studied in many research fields, such as geology [2, 3], oceanography [4, 5] and sedimentology [6, 7]. A very interesting application in crystallography is the potential of the iron carbonate to be used as precursor to prepare Fe_3O_4 and Fe_2O_3 crystals [8] by partial or total oxidation of Fe^{2+} ions to Fe^{3+} ions, respectively.
Continuing our previous studies [9], the present experimental procedure is focused on the hydrothermal synthesis of iron carbonate microparticles in a pure crystalline structure, by hydrothermal decomposition of the Fe(III)-EDTA complex in the presence of urea, starting from Fe(III)-Ferric Ammonium Sulfate (FAS) and Na_4EDTA as main precursors.

Experimental methods
Synthesis
The following procedure of chemical preparation was followed:
An aqueous solution of $1.05x10^{-1}$M of FAS, an aqueous solution of $1.05x10^{-1}$M Na_4EDTA, and an aqueous solution of $9.71 \cdot 10^{-1}$ M of urea were mixed under continuous stirring. This solution was transferred into a number of Teflon-lined stainless-steel autoclaves and was heated up to 230°C and 250ºC by a rate of 1.7ºC/min. The autoclaves were removed, one by one, every two hours in the range between 15 and 26 hours. All the pH measurements indicated a value between 9.4 and 9.5 for the final solutions. The obtained microparticles were filtrated, washed with bidistilled water and dried at 60°C in air.

Results
The crystalline structure of the $FeCO_3$ microparticles synthesized between 15 and 26 hours of high pressure-temperature treatment time is confirmed by XRD analysis spectra (Figures 1.a) in agreement with the respective ICSD (Inorganic Crystal Structure Database) reference code: 01-083-1764. The high purity of FeCO3 microcrystals synthesized between 16 and 24 hours of high

pressure-temperature treatment time is confirmed by EDAX analysis. All the spectra collected in this interval have the same characteristics as the presented spectra (Figure 1.b), which indicates the presence of iron and oxygen only, without any traces of Na, S, C and N, which could result from EDTA and FAS decomposition.

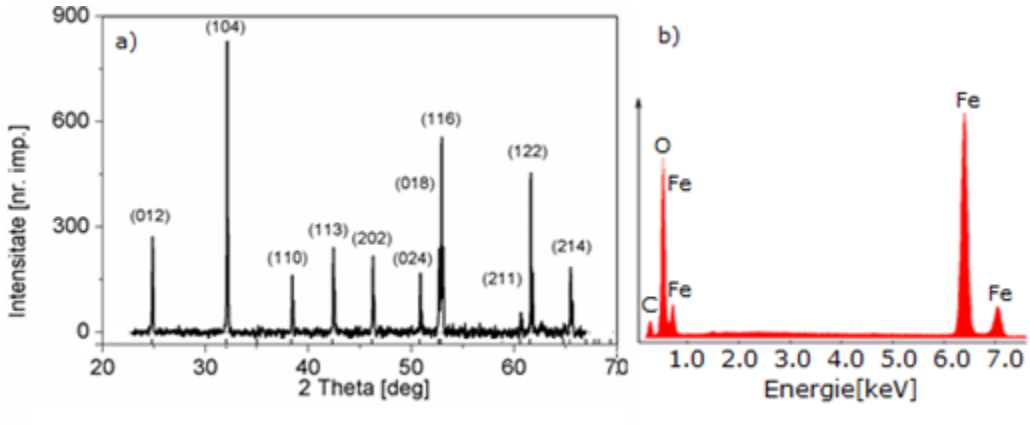

Figure 1. XRD (a) and EDAX (b) spectra.

The SEM images reveal the rhombohedral morphology of the $FeCO_3$ microcrystals synthesized after 22 hours of high pressure-temperature treatment time. The sizes of the $FeCO_3$ microcrystals are in the range of 50 - 200 μm and were evaluated following the SEM image (Figures 2).

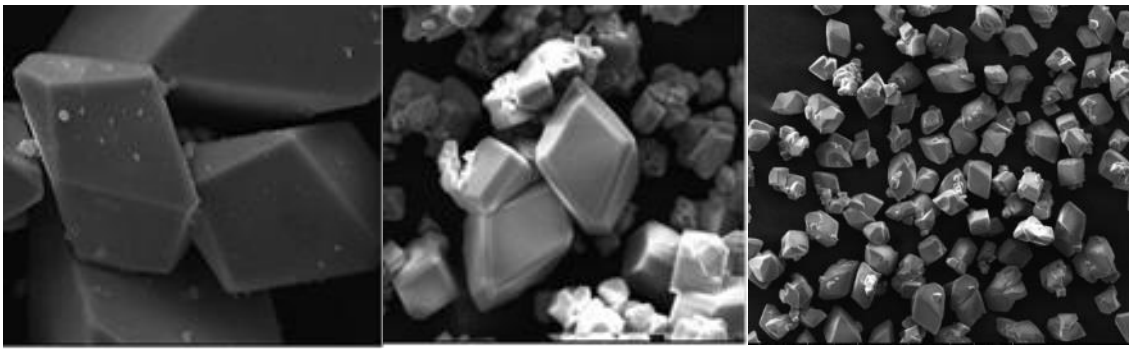

Figure 2. SEM images of the $FeCO_3$ microcrystals

Our preliminary research has shown that by changing some synthesis conditions, e.g. autoclavation time between 15h and 26h and synthesis temperature between 230ºC and 250ºC, particle size may be controlled within the range of 10μm-200μm. For a precise control of the particle's dimension, additional experiments have to be done. An extension of these results has been presented by as in [9].

Conclusion

We developed a new procedure for synthesizing highly crystalline $FeCO_3$ microparticles by hydrothermal decomposition of the Fe(III)-EDTA complex in the presence of urea, starting from Ferric Ammonium Sulfate and Na_4EDTA as main precursors. Single phase $FeCO_3$ microcrystals with sizes in the range of 50μm-200μm were obtained after high pressure-temperature treatment time between 15 hours and 26 hours at 230ºC - 250ºC. The synthesis of pure iron carbonate

microparticles was confirmed using X-ray powder diffraction and EDAX investigation. The present investigation has demonstrated the possibility of synthesizing microsize iron carbonate particles, having rhombohedral morphology, starting from Fe^{3+} ions only and using the hydrothermal decomposition of the Fe(III)-EDTA complex in the presence of urea.

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