Experimental report

16/02/2017

Proposal: 6-05-980 **Council:** 4/2016

Title: Structural characteristics of bioinspired amorphous calcium carbonate: Total scattering using polarized neutrons

Research area: Chemistry

This proposal is a new proposal

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Samples: CaCO3H2O

Instrument	Requested days	Allocated days	From	To
D3	7	9	14/09/2016	23/09/2016

Abstract:

Calcium carbonate (CaCO3) is a widely studied inorganic compound very abundant in nature as a mineral and a biomineral. In the last two decades, many studies have reported the formation of CaCO3 biominerals through an amorphous precursor -Amorphous Calcium Carbonate (ACC)-, which contains structural water. This structural water has been proposed to control the stability of the amorphous structure. However, a clear picture of the structural characteristics of water in ACC is still lacking. This is mainly due to the difficulties associated with the deuteration of ACC. In this study we propose to use polarized neutron scattering to measure the spin incoherent part of the scattering, and thus be able to obtain total neutron scattering data of ACC without the need of deuterating the sample. Different types of water in ACC will be studied by pre-treating the samples at different temperatures following a TGA curve previously measured. This data will be highly valuable to be able to describe the structural characteristics of water in ACC.

ILL Experimental report

Title: **Structural characteristics of bioinspired amorphous calcium carbonate: Total scattering using polarized neutrons**	*Experiment number* **6-05-980**

Experimental team

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Dates: **14/09/2016 to 23/09/2016**

Local contacts: **Gabriel J. Cuello, Anne Stunault**

1. *Goal of the experiment*

Calcium carbonate ($CaCO_3$) is a widely studied inorganic compound very abundant in nature as a mineral and a biomineral. In the last two decades, many studies have reported the formation of $CaCO_3$ biominerals through an amorphous precursor -Amorphous Calcium Carbonate (ACC)-, which contains structural water. This structural water has been proposed to control the stability of the amorphous structure. However, a clear picture of the structural characteristics of water in ACC is still lacking. This is mainly due to the difficulties associated with the deuteration of ACC. In this study we propose to use polarized neutron scattering to measure the spin incoherent part of the scattering, and thus be able to obtain total neutron scattering data of ACC without the need of deuterating the sample. This data will be highly valuable to be able to describe the structural characteristics of water in ACC.

2. *Results*

A pure ACC sample was studied first (Figure 1a), following our list of priorities and as a test for the time needed per sample. This sample was measured for 48h, in order to have adequate statistics for substractions (the separation of the coherent and incoherent part involves data substraction, which increases error bars).

Given the long counting times, we decided to move to amorphous samples with different Mg^{2+} content. This was decided at the expense of measuring ACC samples with lower water contents, which can be measured in D4. Data from a pure amorphous magnesium carbonate and from a mixed Ca and Mg amorphous carbonate (approximately $Ca_{0.5}Mg_{0.5}CO_3$ H_2O) are shown in Figure 1b and 1c, respectively.

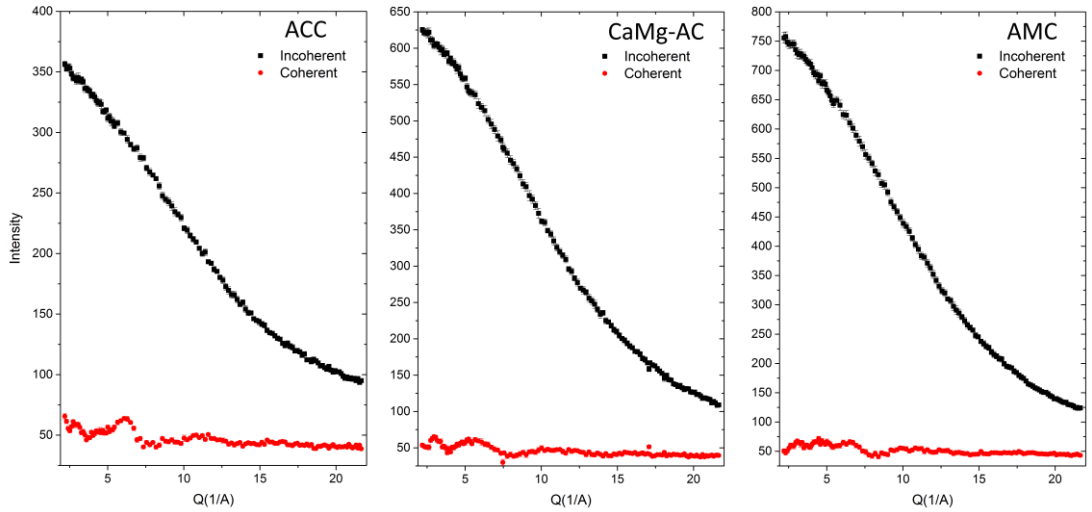

Figure 1. Coherent and incoherent intensity for the three samples under study: ACC, AMC and a mixed CaMg amorphous carbonate.

3. Perspectives

A data analysis strategy is being implemented in which these data are analyzed together with X-ray data taken at ID31 (ESRF) during the same days of the D3 experiment. Reverse Monte Carlo fits of the structure factors obtained here will be performed using both datasets simultaneously.

More experiments will be performed in the future with ACC doped with other ions (polyphosphates, organic acids), that are known to be present in biogenic amorphous carbonates.