

## Report of the experiment n. 08-02-652 on BM08

The report deals about the experiment 08-02-652, performed for 5 days from 08/03/2010 (beam line BM08). We measured diffraction diagrams of 8 different samples of nano-lime suspensions, to be used in Cultural Heritage conservation and obtained by different synthesis methods, and we investigate the kinetics of the carbonatation process in air of three of them.

The nano-limes suspensions were synthesised, at high temperature ( $T=90^{\circ}\text{C}$ ), by a chemical precipitation process starting from supersaturated aqueous solutions of calcium chloride ( $\text{CaCl}_2$ ) and sodium hydroxide ( $\text{NaOH}$ ). In particular, two synthesis methods were considered. The A) method, (suggested by literature):  $\text{NaOH}$  solution, used as precipitator, was added drop wise to  $\text{CaCl}_2$  solution, maintained at high temperature. The B) method was an alternative one, involving use of a non-ionic surfactant. The water content in the all obtained nanolime suspensions was partially substituted with an alcoholic medium (2-propanol), to improve particles disagglomeration and stability. In particular, the syntheses were separately treated, leaving different residual water contents.

First analysing the kinetics of the carbonatation process, we have investigated the phases in the dispersing medium (water, water/2-propanol), arranging a particular sampler that was able to contain the suspension, without exposing it to air. Investigations of blank signals were performed too. The spectra were registered on an image plate with an exposition time of 20 min. The calibration was carried out comparing the results obtained by a standard  $\text{LaB}_6$  powder. We measured diffraction pattern for 8 samples in suspension in different solvents and prepared with different contents of surfactant. See Fig. 1 preliminary results on one sample.

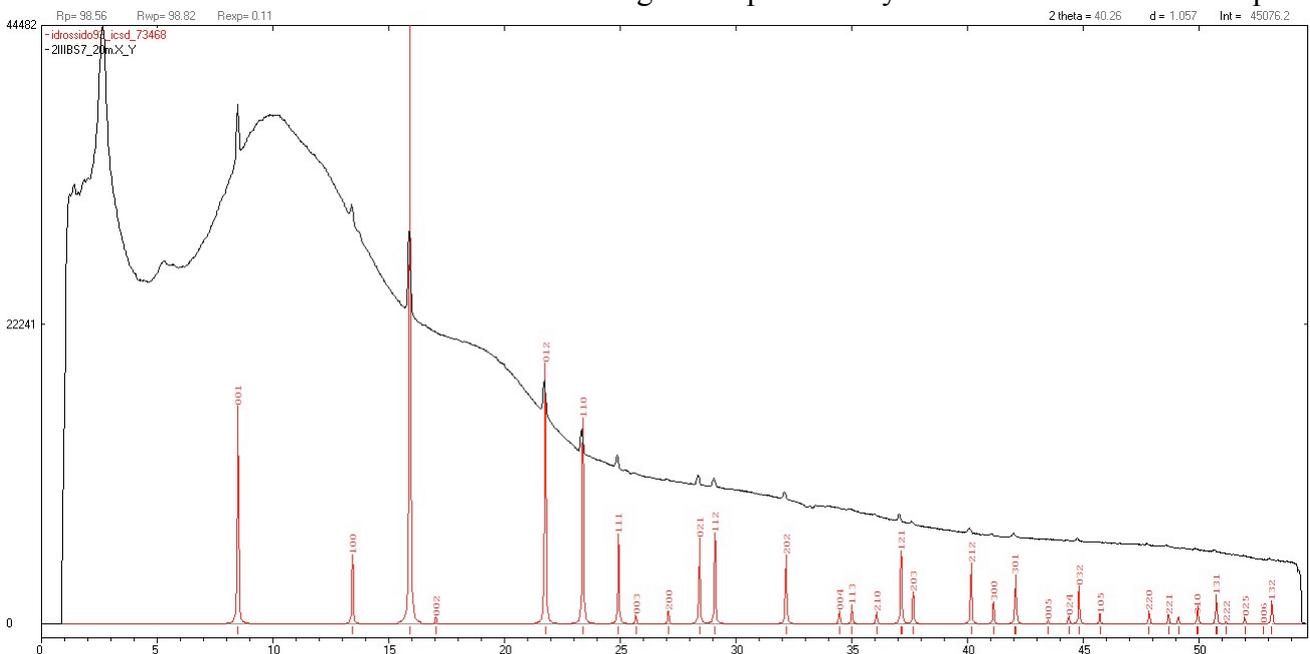


Fig 1. Diffraction pattern of  $\text{Ca}(\text{OH})_2$  nanolimes in suspension (solvent: 50/50 water/2-propanol solution).

In order to investigate the carbonatation process kinetics, we used as substrate pure Whatman cellulose. The investigation were performed depositing a nanolime suspension sample onto the filter paper (Whatman) substrate and leaving it exposed to the X-ray beam until the water/2-propanol was completely evaporate; in particular, time interval of 10 minutes. We measured kinetics on 3

samples and for all of them we got a diffraction pattern on the same samples completely dried. See fig. 2.

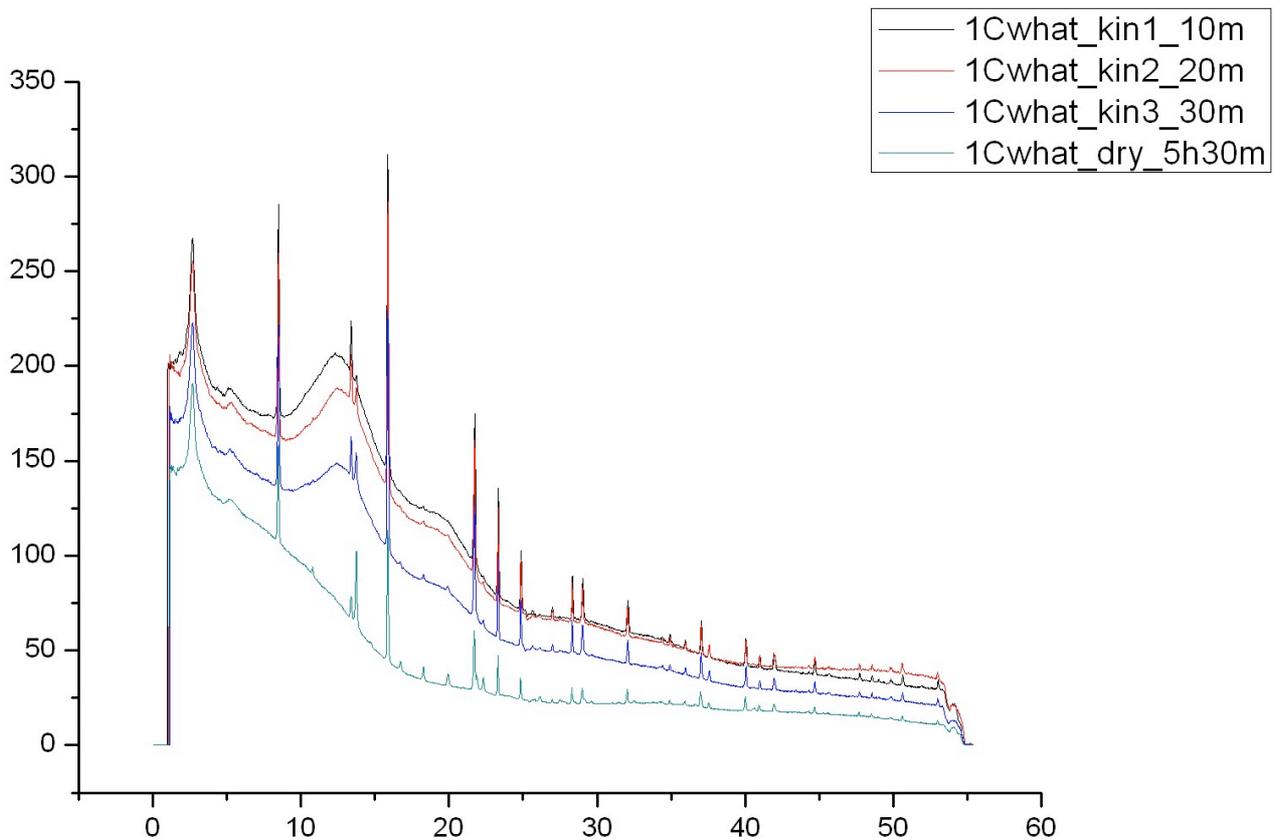


Fig. 2 Monitoring of carbonation process of the nano-limes drying.

We are performing Rietveld analysis on the data using Fullprof program (see fig.3), in order to determine the changes in the structural parameters in the kinetics and changing the preparation method of the samples. See Fig. 3

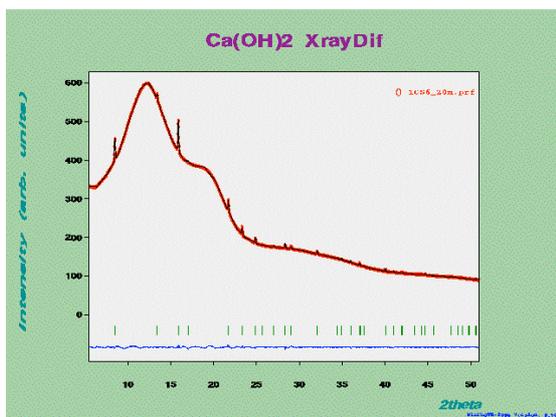


Fig 3. Rietveld refinement of the diffraction pattern of Ca(OH)<sub>2</sub> nanolimes in suspension. In red are reported the experimental data, in black the calculated spectrum and in blue the difference between them.  $\chi^2$