X-RAY POWDER DIFFRACTION

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Introduction

X- ray diffraction (XRD) is one of the most important techniques for characterizing cementitious materials.

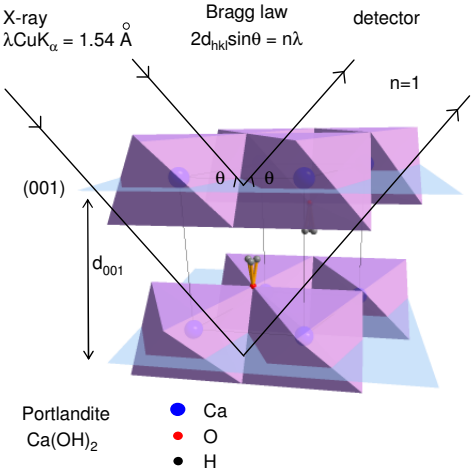
From the XRD pattern, it is generally possible to **identify and quantify crystalline components** which have over 1%wt in a sample.

For quantification, the measured pattern can be fitted to a theoretical pattern generated from data files on the crystal structures of the phases (Rietveld analysis).

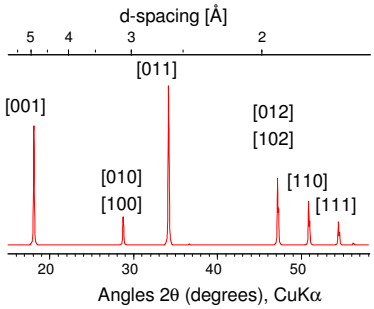
Quantification of amorphous phase may be also possible by adding an internal standard in the powder sample or using an external standard.

Measurement Principle

Experiments



Output for powder



- comparison with the Powder Diffraction File (PDF) database
- identification of the crystalline phases in the powder sample

Relevance for Our Field

Mineralogical quantification of cement is necessary to predict the performances of the resulting concrete.

Kinetics of hydration of the cement phases can be followed as well as the kinetics of formation of the hydrated products. However quantification of the amorphous content is needed.

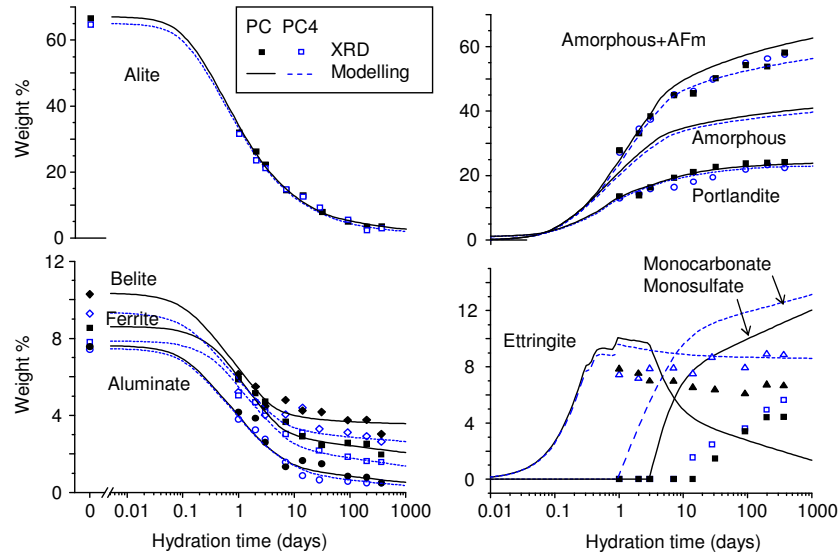
main crystalline phases in anhydrous Portland cement:
- alite Ca_3SiO_5
- belite Ca_2SiO_4
- aluminate $Ca_3Al_2O_6$
- ferrite $Ca_2Al_xFe_{(1-x)}O_5$

main crystalline hydrated products
- portlandite $Ca(OH)_2$
- ettringite $Ca_6[Al(OH)_6]_2(SO_4)_3\,26H_2O$

ill crystallized hydrated products
- monocarbonate $[Ca_2Al(OH)_6]\,(CO_3)_{0.5}\,2.5H_2O$
- C-S-H $Ca_x\,Si_y\,zH_2O$

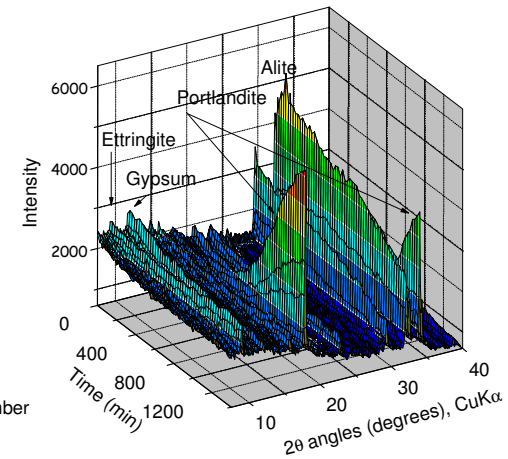
Example

Hydration of an Ordinary Portland Cement without (PC) and with 4%wt of calcite (PC4) [1]



Applications & Potentials

- Identification of crystalline components
- Quantification of the crystalline phases in anhydrous cement
- Quantification of the crystalline phases as a function of hydration time
- Quantification of the amorphous content in hydrated cement paste
- In situ experiments using the Thermal Humidity Chamber



Limitations

- No information on the nature of amorphous phase(s)
- Problem of solid solution
- Difficult to assess an error bar

[1] B. Lothenbach, G. Le Saout, E. Gallucci, K. Scrivener, Influence of limestone on the hydration of Portland cements, Cement and Concrete Research 38 (2008) 848-860.