

Overview of basic chemical and pore structure analysis for binder reactions

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THERMAL ANALYSIS

Thermogravimetry (**TG**)

- mass as a function of temperature or mass as a function of time at constant temperature
- first derivative, i.e. rate of mass change (**DTG**)

Differential thermal analysis (**DTA**)

- energy liberation/absorption connected to mass changes (exothermic/endothemic)

Sample

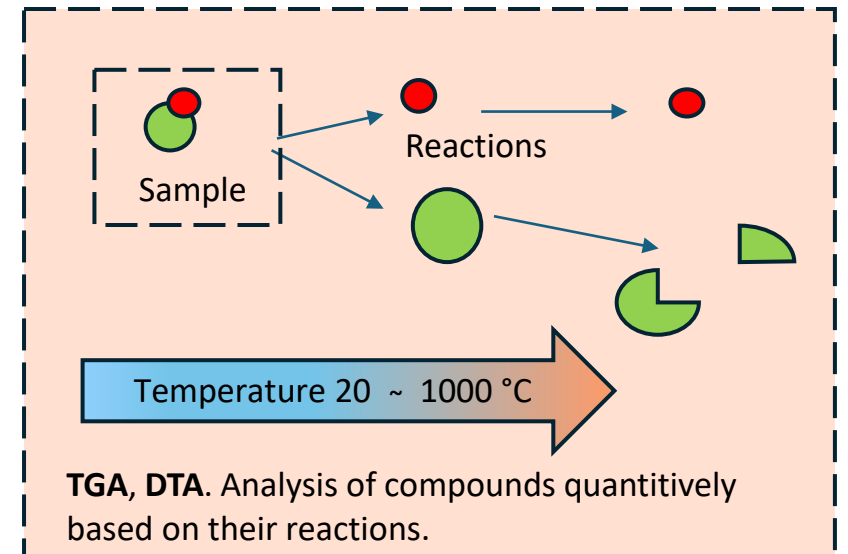
- powder, solid or liquid, size 1 mg - 5 g (usually 50 - 200 mg)

Applications

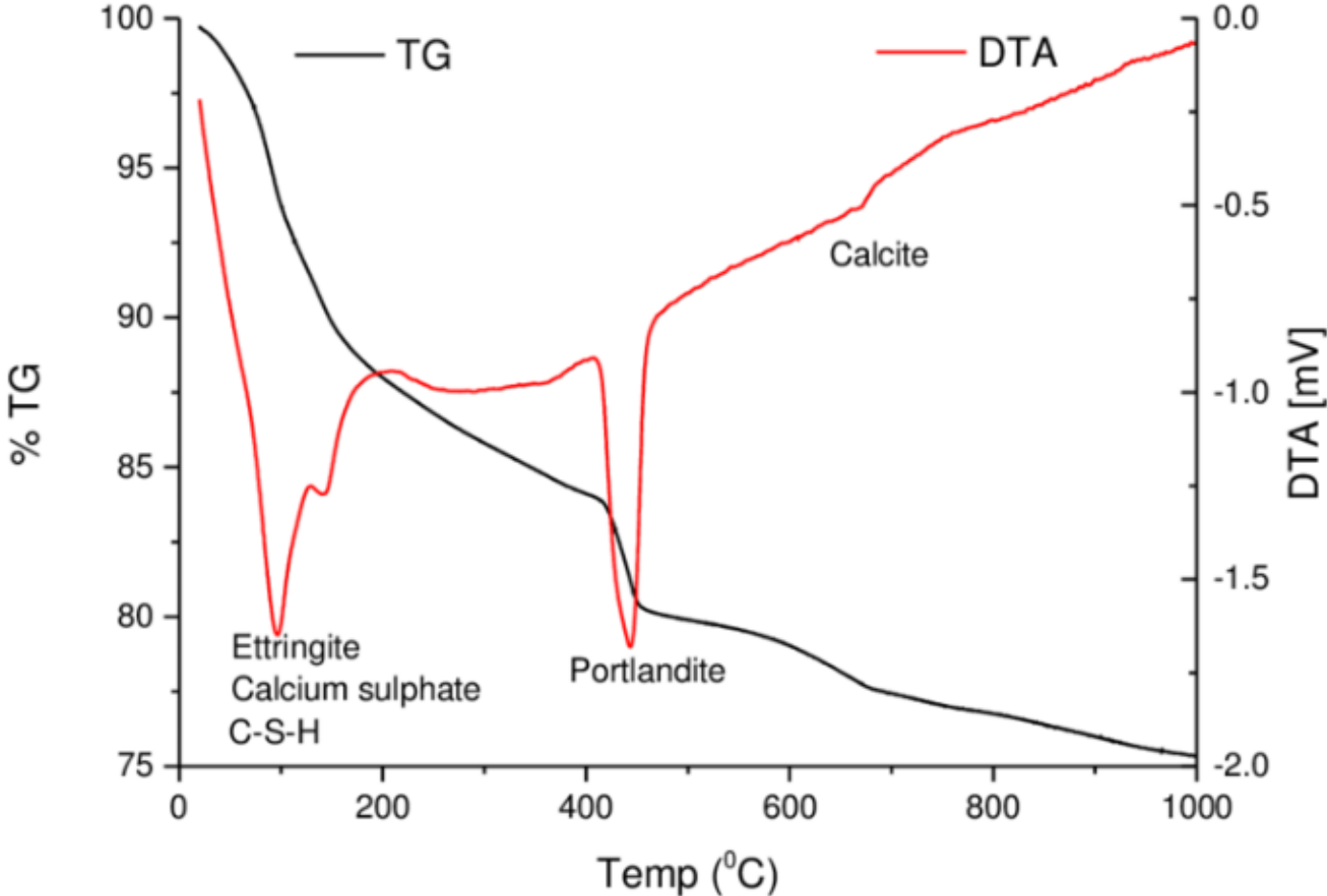
- amount of bound water
- reaction degree of cement and slag (**DTA**)
- - quality of hydration products

Ca(OH)₂ and CaCO₃ content os pasre

Other useful methods **DSC**, dilatometry, **TMA**, **DMA**



THERMAL ANALYSIS, TG and DTA



5: Typical TGA and DTA curves for a hydrated cement paste

X-RAY DIFFRACTION (XRD)

Identification of crystalline compounds

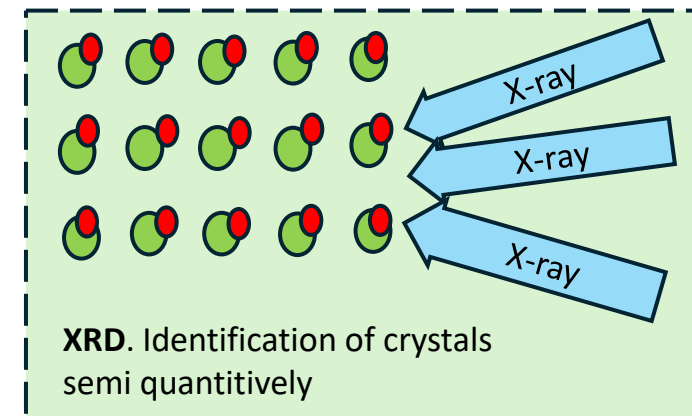
- intensities of diffracted X-rays as a function of angle with respect to the incident beam
- distances between the atoms of the compounds of the material
- "fingerprint" of the compounds, data file identification
- mostly qualitative

Sample

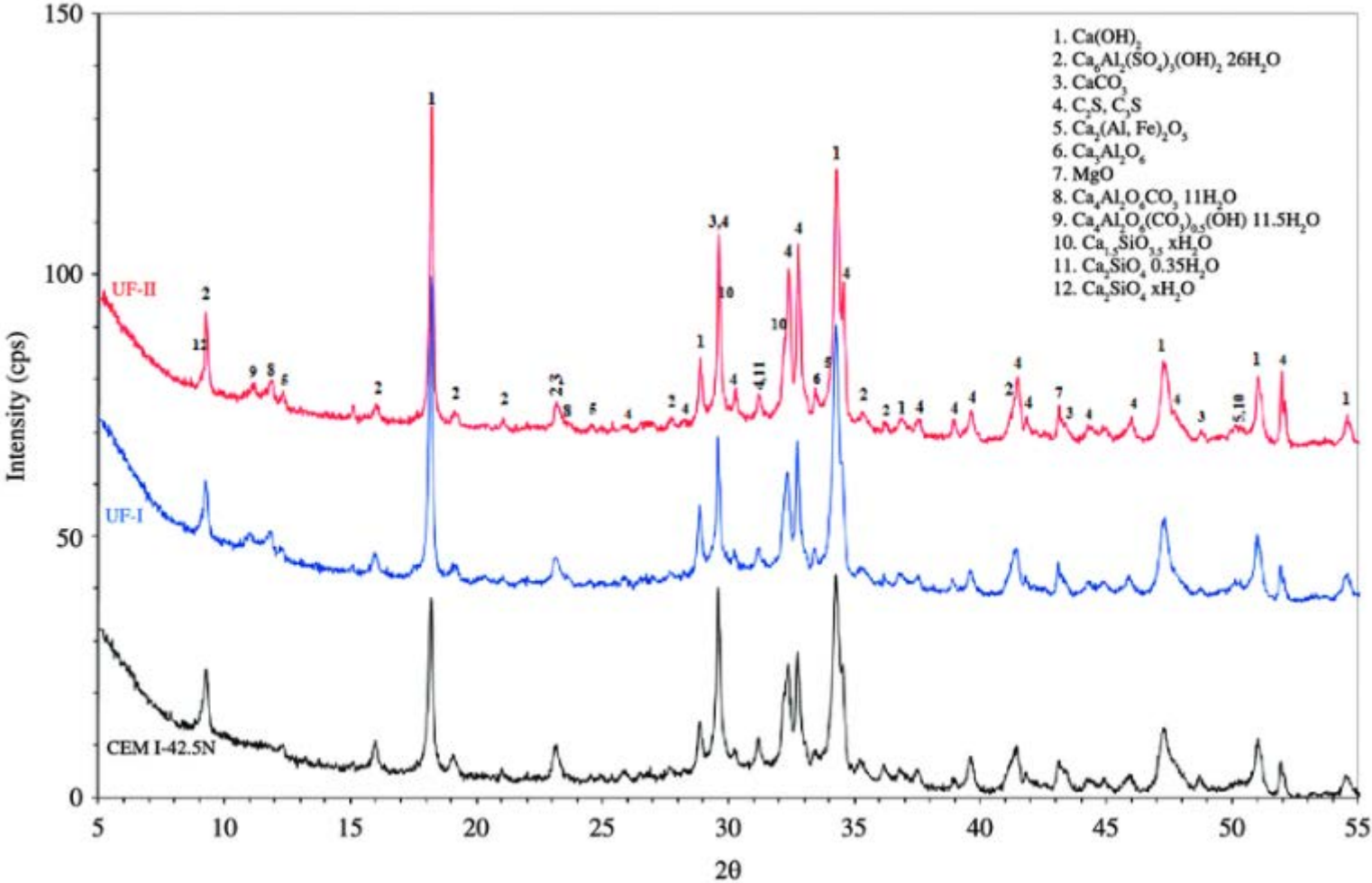
- powder, solid, (liquid), film, surface, size from a few milligrams to a few grams, restrictions regarding dimensions

Applications

- mineral composition
- clinker
- calcite, portlandite, ettringite, gypsum, other salts, residual clinker minerals



X-RAY DIFFRACTION (XRD)



X-Ray Diffraction of tested cements hydrated at 1 day.

X-RAY FLUORESCENCE (XRF)

Analysis of element contents

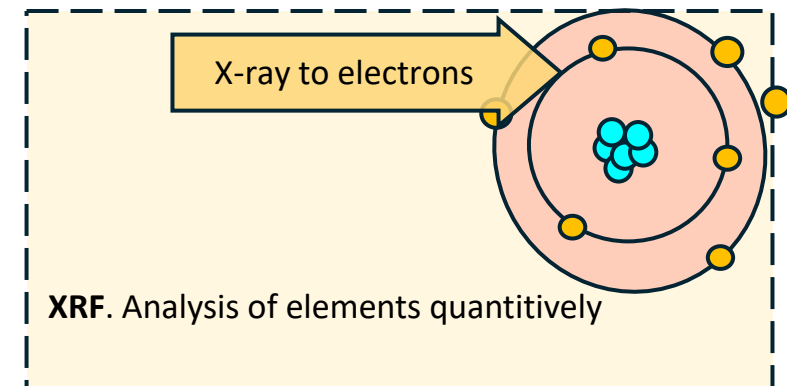
- emission of secondary X-rays from a material excited by incident X-ray radiation
- energies (**EDS**) and wavelengths (**WDS**) of emitted X-rays specific to each element, identification
- emitted intensities, quantification

Sample

- powder, solid, (liquid), film, surface, size from a few milligrams to a few grams, restrictions regarding dimensions

Applications

- element composition (no light elements, **TG/XRD** often needed)
- surface analysis



ENERGY/WAVELENGTH DISPERSIVE SPECTROMETRY (**EDS/WDS**)

Element analysis in connection with scanning electron microscope (**SEM**)

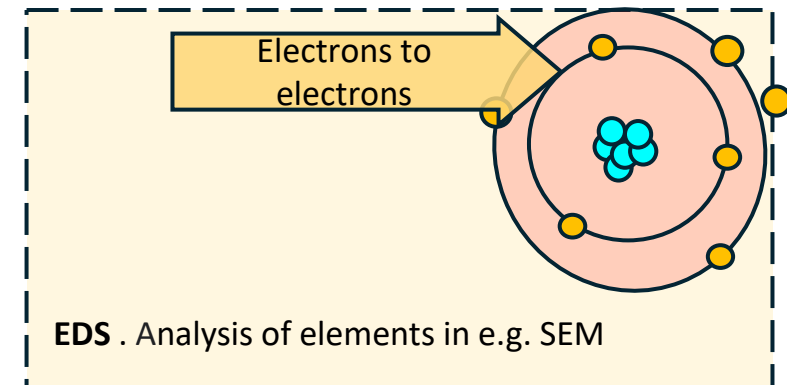
- as **XRF** but excitation by electrons

Sample

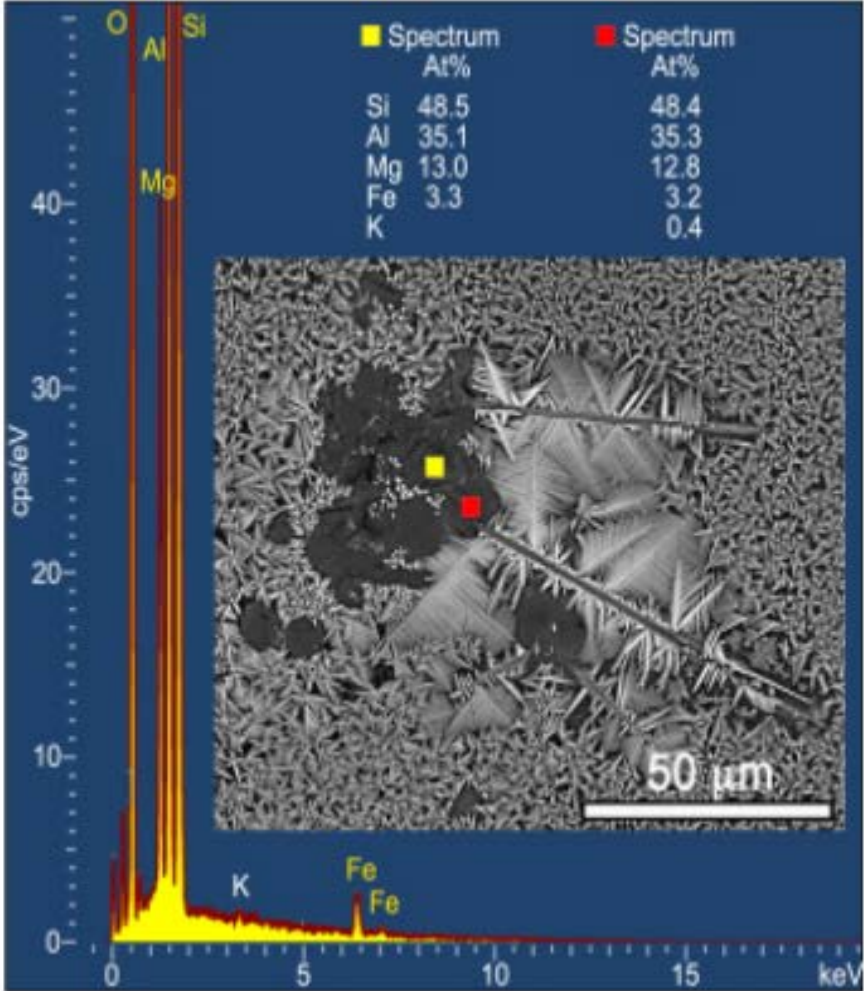
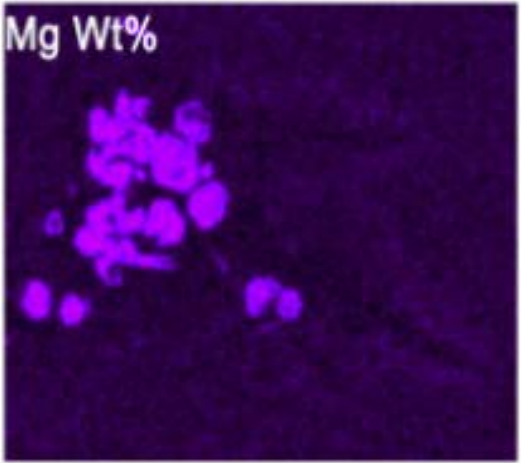
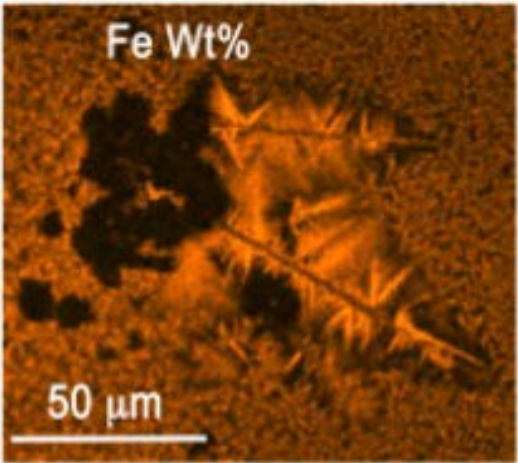
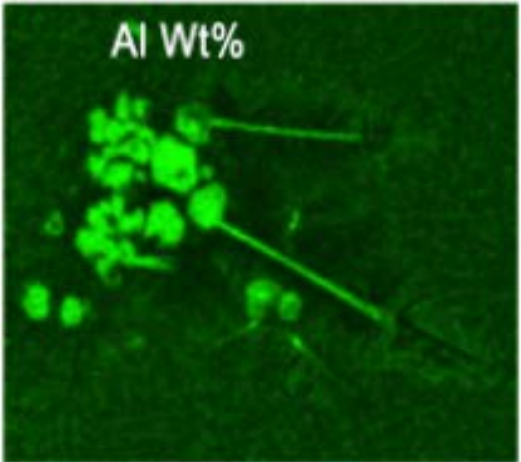
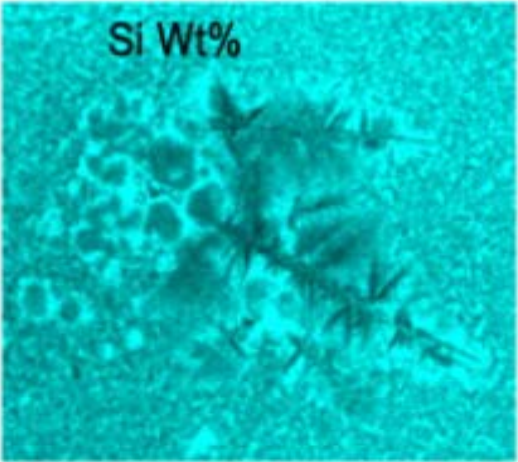
- (dry) powder or solid, restrictions regarding dimensions

Applications

- details and images of structure (**SEM**)
- element analysis
- spot analysis, line analysis
- element maps, distribution of elements
- materials in pores (ettringite, **ASR** gel)



ENERGY/WAVELENGTH DISPERSIVE SPECTROMETRY (EDS/WDS)



FOURIER TRANSFORM INFRARED SPECTROSCOPY (**FTIR**)

Identification of organic compounds

- absorption of IR radiation of the material due to the functional groups of the compounds
- "fingerprints" of the compounds, identification by a data file
- equivalent to **XRD**
- mostly qualitative

Sample

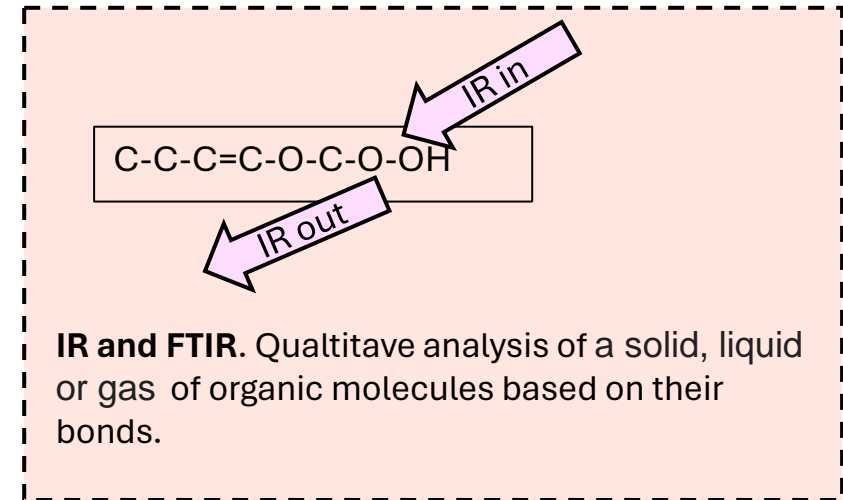
- powder, solid, liquid, film surface, size from a few milligrams up

Applications

- identification of organic compounds

Other methods for quantitative and more accurate (eg. binder additives) analysis available

- mass spectrometry (**MS**)
- liquid (**LC**) or gas (**GC**) chromatography



FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

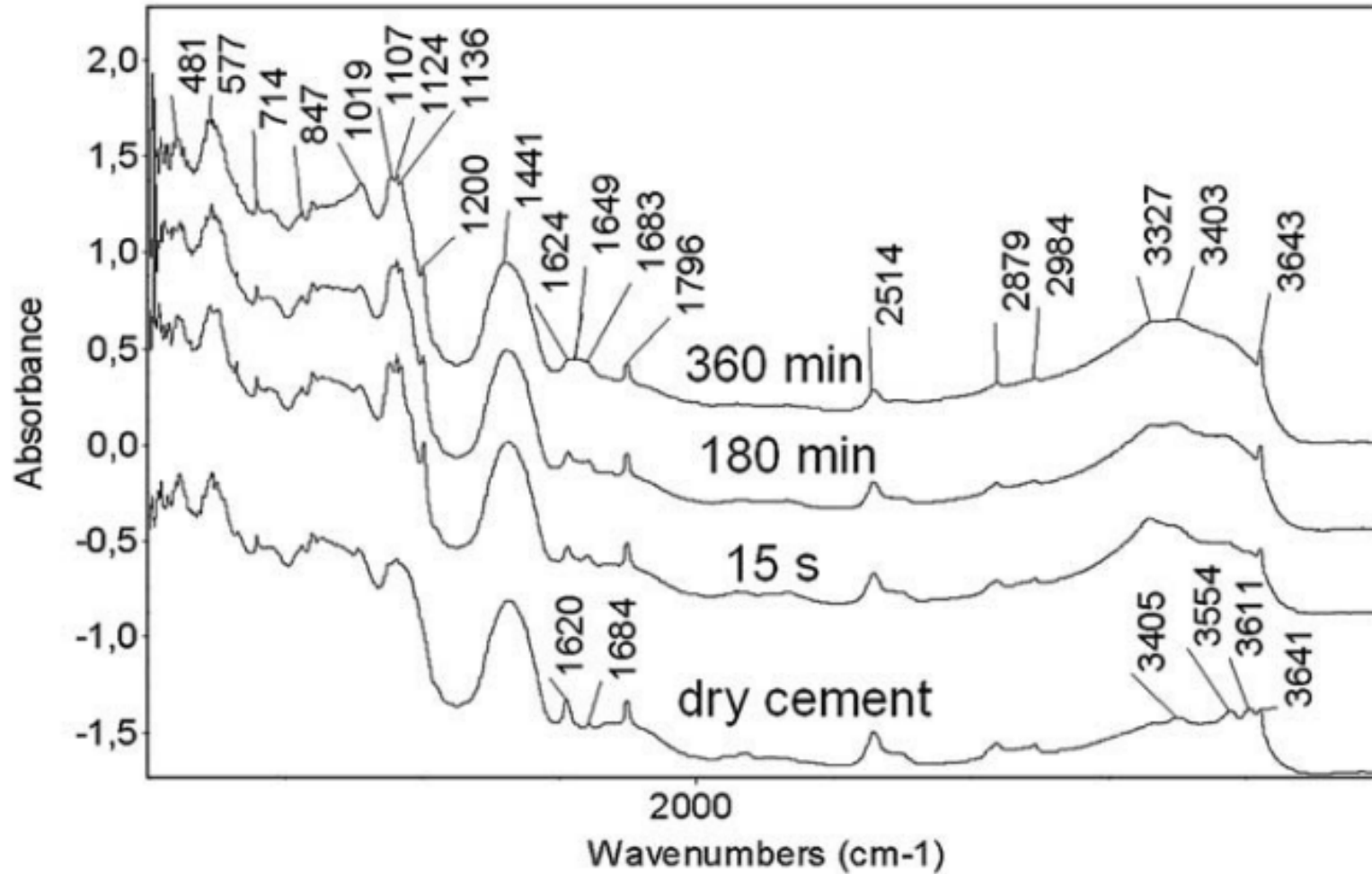


Fig. 2. Absorbance of as received dry cement and cement that has been allowed to hydrate for 15 s, 180 min and 360 min after the cement was added to the water. The spectra are shown offset for clarity.

OTHERS

Several methods for special purposes

- **PIXE, AES, XPS, RBS, MS**

Nuclear magnetic resonance (**NMR**)

- interaction of nuclei of certain atoms, eg. hydrogen, exposed to a strong magnetic and **RF** fields
- information on the chemical bond patterns and on the stereochemical arrangements
- especially for the study of the structure of amorphous materials like hydrated cement and some polymers

Wet chemical methods

- The structure is dissolved → Destroyed
- soluble compounds (chloride, sulphate)
- special applications (ground water, **ASR**)

Thin sections

- structural details, cracking
- pore structure

