CONDUCTION CALORIMETRY
Thermometric TAM Air

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Introduction

The isothermal (heat conduction) calorimetry is an efficient tool to study the stages related to the hydration of cement pastes or mortars at constant temperature. The calorimeter continuously **measures and displays the heat flow related to the hydration reactions** taking place in the cement paste after mixing (Fig. 1).

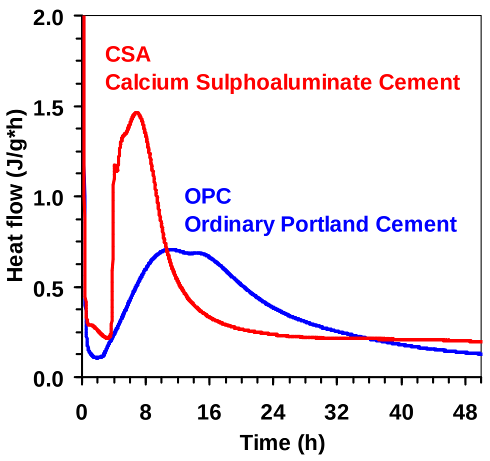
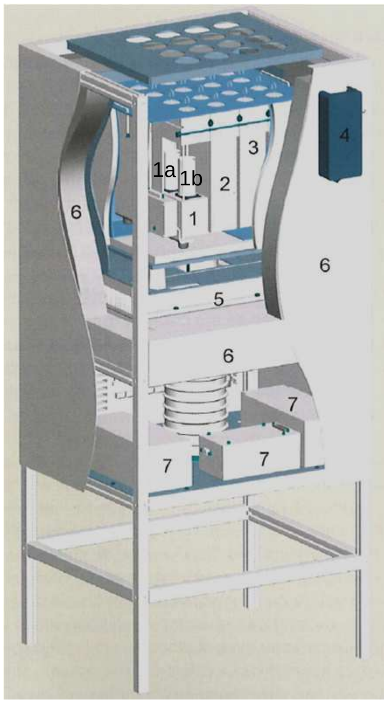


Figure 1: Heat flow related to the hydration of OPC and CSA cements.

The Instrument

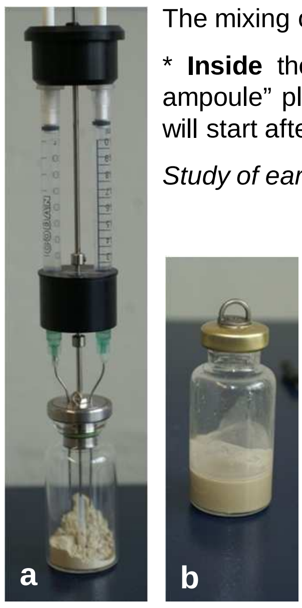


Thermometric TAM Air instrument:

* 8 twin calorimeters (sample + reference reduces noise level)
* High sensitivity (± 10 µW)
* Good stability (long-term fluctuations < 20 µW over 24h)
* Long-term analyses possible (2 weeks)
* Small sample volume (~1-2 g of cement paste)
* Calorimeters in an air thermostat (Peltier heater/cooler)
* Temperature range 5-90 ℃
* Modes ± 60 mW (long-term analysis) or ± 600 mW (early hydration)

Figure 2:
Cut-away drawing of the instrument. 1-3: twin calorimeters (a: reference, b: sample), 4: temperature regulator, 5: Peltier heater/cooler, 6: insulation, 7: amplifier, data logger, power supplies. Modified from Wadsö (2005).

Mixing techniques



The mixing of the dry cement/mortar with water can be done:

* **Inside** the calorimeter: sample preparation in an "Admix-ampoule" placed in the calorimeter. Mixing and measurement will start after ~1.5h after thermal equilibration.

Study of early hydration reactions.

* **Outside** the instrument: mixing in a glass ampoule closed with a Teflon coated septa. The ampoule is placed after mixing in the calorimeter.

Late hydration reactions (first 30 min not usable).

Figure 3:
Ampoules of 20 ml prepared for analysis. a: "Admix-ampoule" with two syringes of 1 ml for mixing in the instrument, b: normal ampoule with septa.

An innovative technique

Coupling calorimetry and expansion measurement

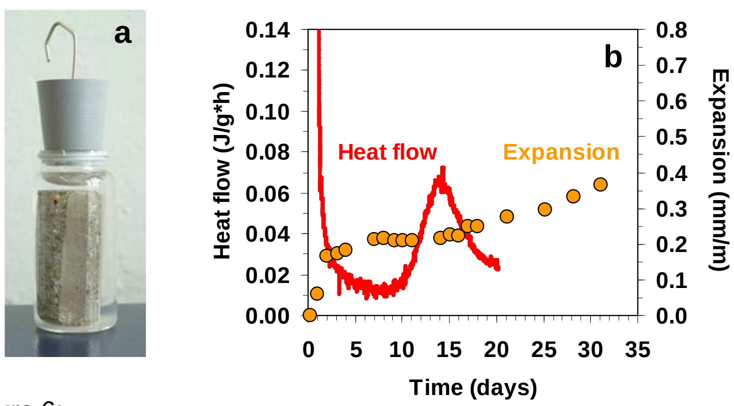


Figure 6:
Study of late expansion potential of some rapid-hardening mortars. a: mortar prism in the ampoule filled with water, b: results of long-time calorimetric and expansion measurement.

Applications

Industry-related

* Determination of total heat
* Setting behavior
* Quantifying retardation/acceleration
* Quality control on cement plants
* Optimization of calcium sulphate addition
* Influence of calcium sulphate type

Research tool

* Very early hydration reactions
* Measurements at different temperatures
* Quantifying retardation/acceleration
* Influence of contaminants
* Proportioning of cement based products
* Hydration mechanism of pure components

Examples

Quantifying retardation

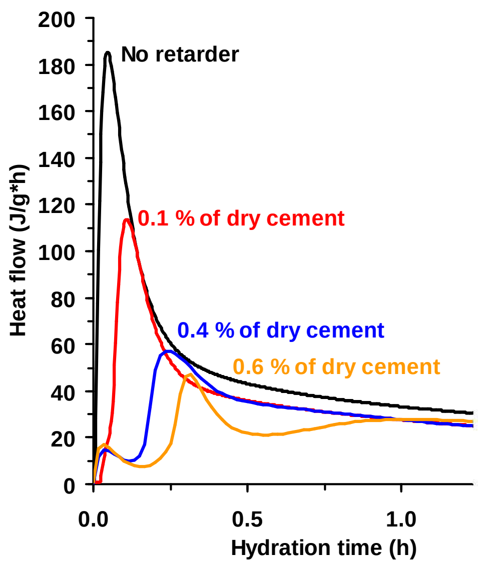


Figure 4:
Addition of retarder to a cement used for the preparation of a rapid-hardening mortar.

Gypsum addition

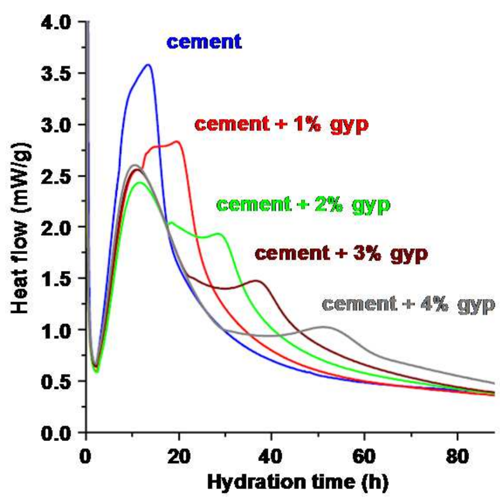


Figure 5:
Gypsum addition to an OPC-slag cement. Modified from Kocaba (2009).

Limitations

* No information about the solids:
  Ideally coupled with XRD (type), TGA (quantity)
* Long-term measurement:
  Problem of baseline stability
* Dedicated to mortar with grain sizes < 4 mm (larger aggregates do not give representative results)

References

Kocaba V. (2009). Development and evaluation of methods to follow microstructural development of cementitious systems including slags. PhD Thesis, EPFL, Switzerland, 235 p.

Wadsö L. (2005). Applications of an eight-channel isothermal conduction calorimeter for cement hydration studies. Cem. Int. 5, 94–101.