Laboratory of Construction Materials

Isothermal Calorimeter

Wilasinee HANPONGPUN and Karen SCRIVENER
Laboratory of Construction Materials, École Polytechnique Fédérale de Lausanne (EPFL), Switzerland

What is it used for?

- ❑ **For measuring heat releases from the hydration process of cement** [1]
  - The shape of the heat flow versus time curve reflects the hydration process(es) of cement
  - The integrated heat flow time curve, i.e. the energy evolved is related to the extent of hydration

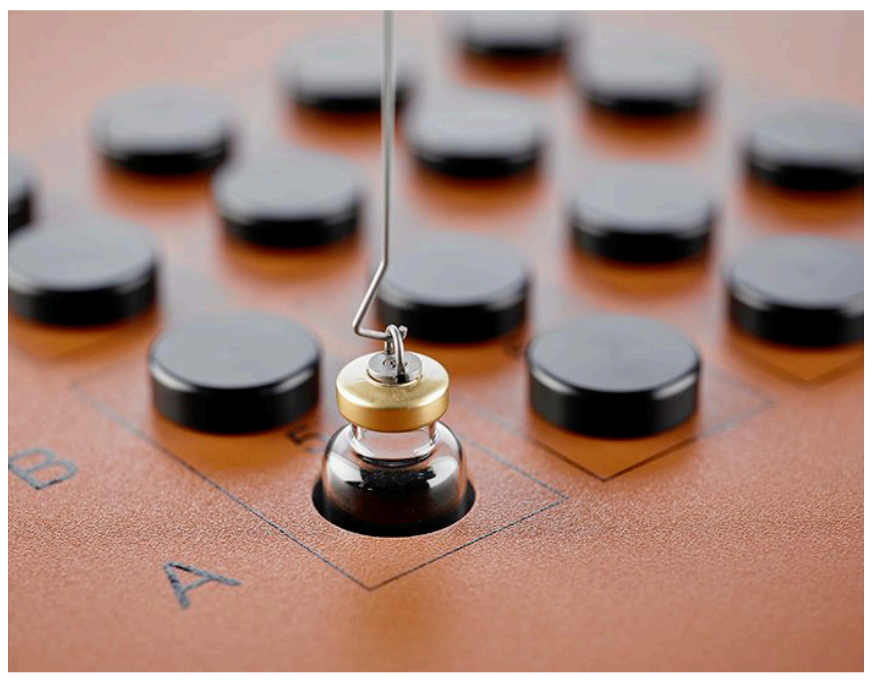


Figure 1. TAM Air Isothermal calorimeter [2]

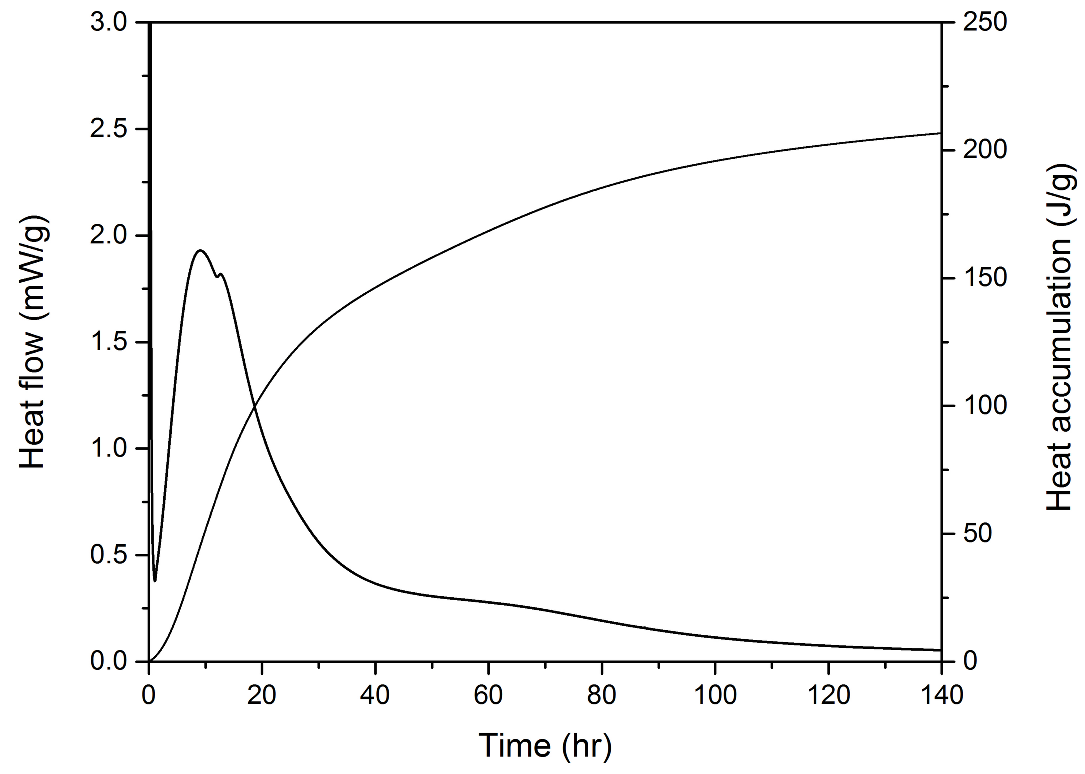


Figure 2. Heat flow and heat accumulation of OPC type I

Applications

- ❑ **Hydration of cementitious materials**
- ❑ **Setting time and stiffening**
- ❑ **Effect of admixtures**
- ❑ **Effect of temperature**
- ❑ **Dosing and formulation optimization**
- ❑ **Quality control**
- ❑ **R&D**

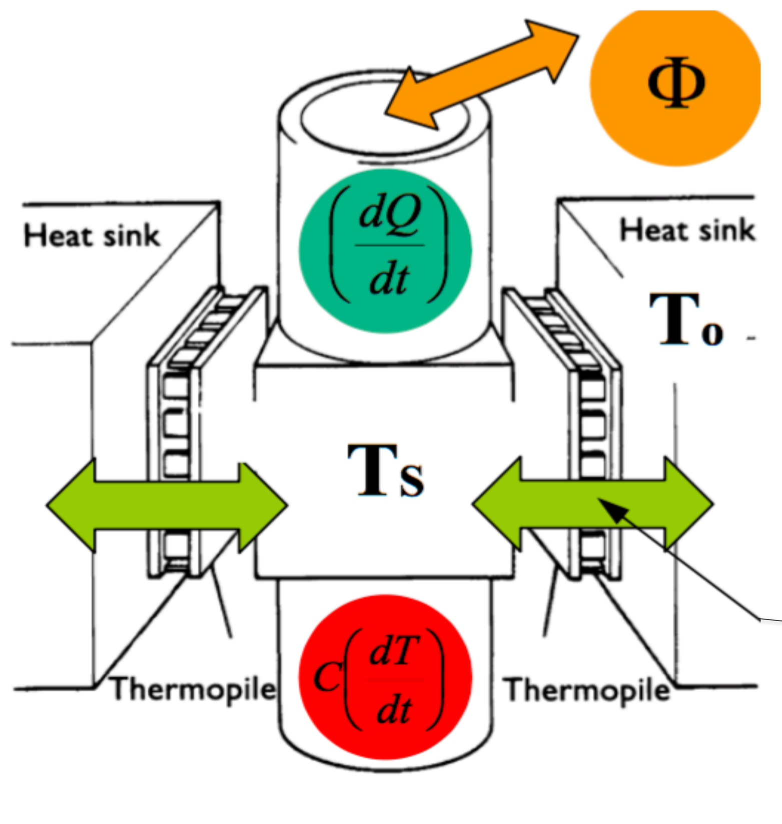


Figure 3. Calorimetric unit [3]

General heat balance equation

$$\frac{dQ}{dt} = \Phi + C\left(\frac{dT}{dt}\right)$$

Rate of Heat Production = Rate of Heat Exchange + Rate of Heat Accumulation

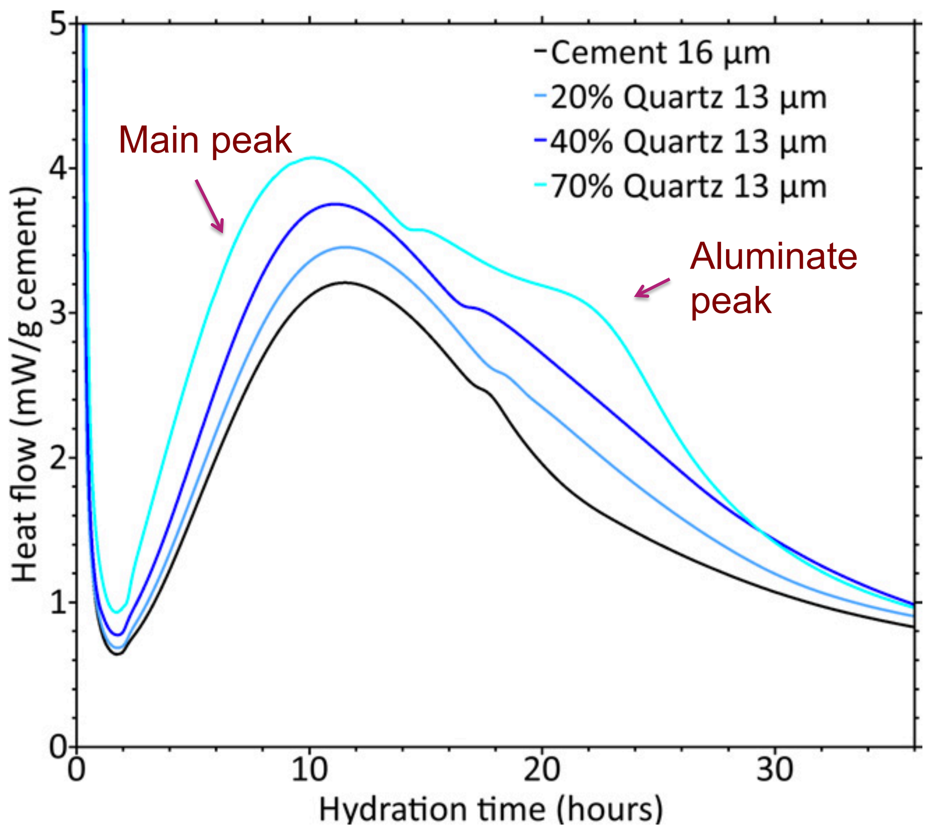
The measured property

Heat flow monitored by TAM Air = Rate of heat production (dQ/dt)

After calibration the following holds

Examples

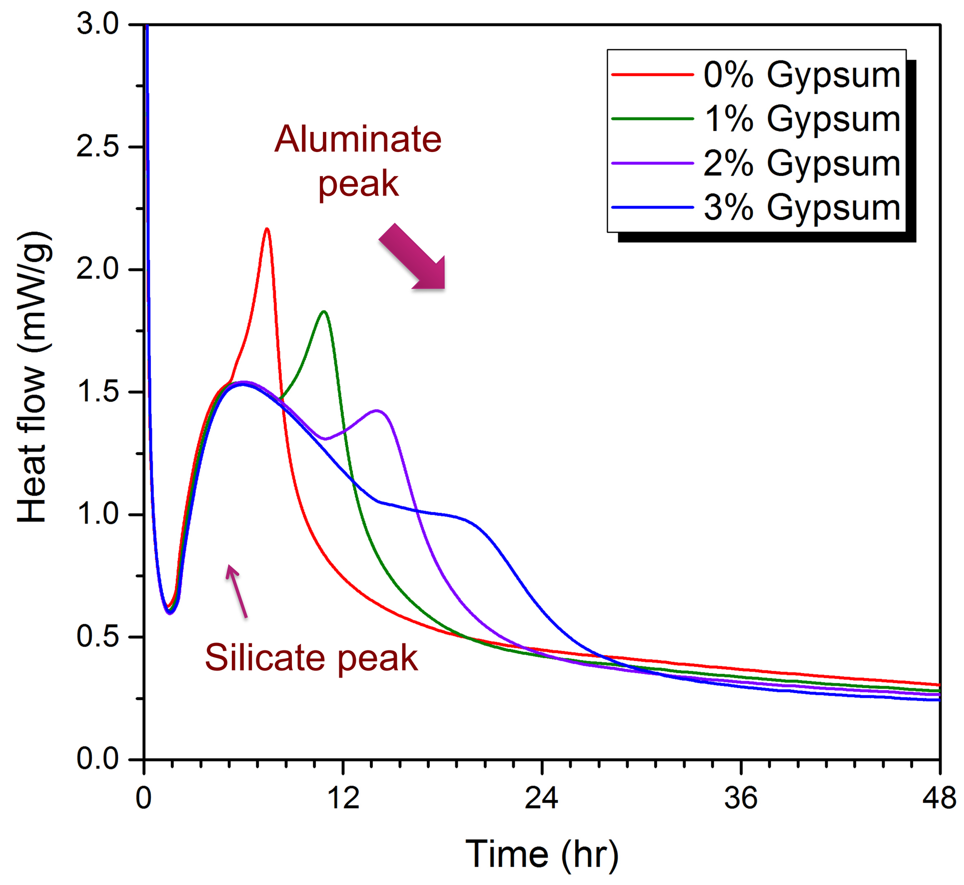
❑ Filler effect [4]



The replacement level of quartz ranged from 20% to 70%. Although quartz is inert, the rate of reaction of the clinker component is enhanced. The main peak and aluminate reaction are accelerated. This effect resulted from the SCM the physical replacement called "filler effect".

Figure 4. the heat flow curves of hydration for quartz–cement pastes, normalized by the plain Portland cement content

❑ Gypsum adjustment



Without gypsum adjustment, silicate and aluminate peaks occur close to each other, leading to a lower total heat release and reactivity. So, a proper gypsum addition in this case is 1%. It is possible to differentiate the two peaks from each other. This ensures the optimal properties of the blended cement.

Figure 5. the heat flow curves of LC^3-50(2:1) blended cement with different gypsum content, normalized by cement paste

❑ Activation energy (Ea)

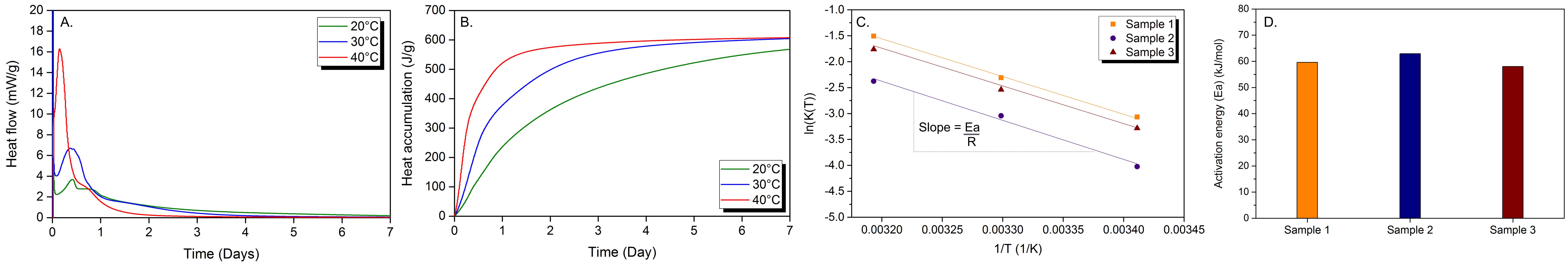


Figure 6. The determination of the activation energy calculated from Arrhenius equation using the concept of equivalent time [5]

A. The heat flow over time and directly reflects the rate of reaction for three different temperatures of the same system
B. The integral of this heat flow over time, the total heat for three different temperatures of the same system
C. The determination of Ea from plot of ln(k) vs 1/T of three systems [R = gas constant (8.314 J/mol/K)]
D. Activation energy of three systems calculated from the slope of ln(k) vs 1/T x R for each system

Notes Room MXG239, wilasinee.hanpongpun@epfl.ch

References
[1] http://www.cptechcenter.org/ncc/TTCC-NCC-documents/F2008-F2011/09IsothermalCalorimetry_000.pdf
[2] http://www.tainstruments.com/dt_gallery/tam-air/
[3] http://mech.fsv.cvut.cz/~nemecek/teaching/dmpo/TAM%20Air%20-%20Presentation%20for%20University%20Prague.pdf
[4] E. Berodier, K. Scrivener, Understanding the Filler Effect on the Nucleation and Growth of C-S-H, Journal of the American Ceramic Society, 97 (2014) 3764-3773.
[5] J.L. Poole, K.A. Riding, K.J. Folliard, M.C.G. Juenger, and A.K. Schindler, Methods for Calculating Activation Energy for Portland Cement, ACI Materials Journal, 104 (2007), 303-311.