The Study of Cement Hydration by Isothermal Calorimetry

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INTRODUCTION

Cement is a finely ground powder of burned limestone. It reacts readily with water (hydrates) to form a solid material, known as hardened cement paste. When cement is mixed together with rock aggregate, sand and filler materials, it forms concrete. Cement is one of the most important base materials in general use in the construction industry. The optimisation of concrete with respect to frost resistance, durability, chemical resistance etc. is of great importance.

Cement hydration is a strongly exothermal reaction that takes place in a number of stages (Young 1985):

I. Rapid initial processes
II. Induction (dormant) period
III. Acceleralion period
IV Retardation period
V. Long-term reactions

Isothermal ealorimetry is probably the best way to follow all stages of the hydration process (Bensled 1987). The measurements presented below were designed to demonstrate the hydration in stages II-IV. To study stage I, it is necessary to mix water and cement inside the sample ampoule within the calorimeter. This is quite easy if there is a high water-cement ratio (w/c), however for lower, more realistic water content this is a more difficult experiment (Wadsö 1995). For the long-term reactions of stage V, a microcalorimeter would have to be used to achieve the required increase in detectability.

MATERIALS AND METHODS

Two experiments were performed with TAM Air using glass ampoules. In the first experiment, all eight calorimeters were loaded with 3.40 - 5.61 g of a cement paste containing an additive with retarding properties. In the second experiment, another cement paste with six different concentrations of an additive was tested. The sample sizes were between 4.99-5.85 g. The experiments were performed as a delivery acceptance test at an industrial laboratory; therefore the cement type, w/c or type of admixtures can not be disclosed.

RESULTS AND DISCUSSION

Figures 1 and 2 show the result of the measurements on twelve samples of identical composition. It can be seen that the spread of the results is very low.

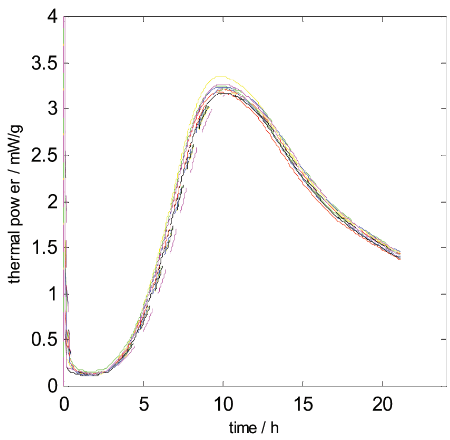

Figure 1. The primary result from twelve measurements of samples of the same cement paste

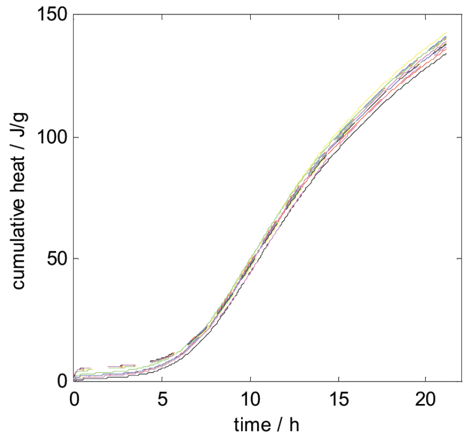

Figure 2. The integrated result from twelve measurements of samples of the same cement paste

Figure 3 shows the result of the measurement of the hardening of a mortar with six different concentrations of an additive. Note that it is possible to see double peaks for the lower concentrations, but not for the higher ones. The two peaks are probably from the main reactions of the two main components (C 3 S, C 3 A). It can be seen from figure 3 that the TAM Air calorimeter is an excellent instrument for the study of retardation.

Adiabatic and semi-adiabatic calorimeters are commonly used in cement and concrete research. For cement paste and mortar measurements, the isothermal calorimeter has been shown to be a better instrument than an adiabatic calorimeter as it gives quantitative data with very high resolution. The isothermal calorimeter is calibrated to give a thermal power output, instead of a quite arbitrary temperature increase as in the semi-adiabatic calorimeter. The thermal power resolution of the TAM Air calorimeter is in the order of 2.5 mW. The only limitation is the sample size, which is a maximum of 20 ml. This is because the heat produced has to be conducted away from the sample during the measurement. It is therefore not advisable to use the TAM Air calorimeter with aggregates larger than 5 mm.

FURTHER EXPERIMENTS

The TAM Air calorimeter is primarily an instrument for the study of the induction period and the main stages of the hydration. Interesting applications include the influence on hydration of glass fillers, waste products, slags and other materials often used in concrete. It should be possible to assess the activation of such materials and to evaluate their effect on the hydration.

The heat production is a result of the hardening processes in the cement paste. It has been shown that the degree of hydration, the heat produced and the strength are related (Taplin 1959, Byfors 1980). Isothermal calorimetric measurements can therefore form the basis of design criteria for the strength and temperature development in concrete structures, as for example, in the minimisation of crack development.

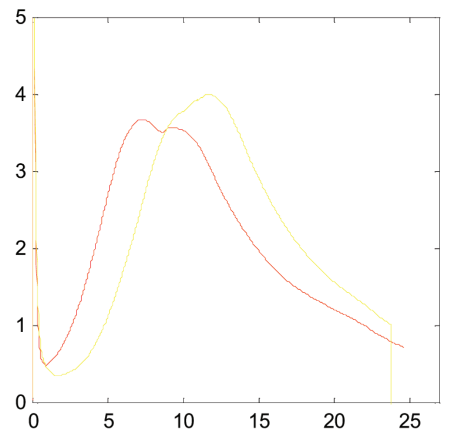

Figure 3. The primary result from twelve measurements of samples of the same cement paste, but with six different concentrations of an additive (0, 0.10, 0.15, 0.20, 0.30 and 0.50%, two samples of each concentration were tested).

REFERENCES

1. Bensted, J. (**l987**) "Some applications of conduction calorimetry 10 cement hydration", *Adv. Cement Res*. I 35-44
2. Byfors, J. (**1980**) "Plain concrete at early ages" *Cement- och Betonginstitutet*, Stockholm, Sweden.
3. Taplin, J M (**1959**) "A method for following the hydration reaction in Portland cement paste"
4. Wadsö, L. (**1995**) "Evaluation of isothermal calorimelry for characterization of very early and early cement reactions, a critical literature study", *Building Materials*, Lund University, Sweden TVBM-7094.
5. Young, J. K (**1985**) "Hydration of Portland cement" in "*Instructional modulus in cement science*", ed. D. M. Roy, Materials education Council. Materials Research Laboratory, University Park, PA. USA