Setting time estimation and prediction using isothermal calorimetry

Instruments to which this note applies: All Calmetrix I-Cal isothermal calorimeters

Target use: Setting time estimation, materials selection, mix design optimization, admixture formulation, quality control in cement manufacturing, dry mortar

Introduction

Isothermal calorimetry is very well suited for set time estimation or even prediction given its precise temperature control. For most concrete, setting occurs shortly after the end of the dormant period B when the hydrate formation accelerates to cause setting, as indicated by the vertical lines representing initial and final set in Figure 1.

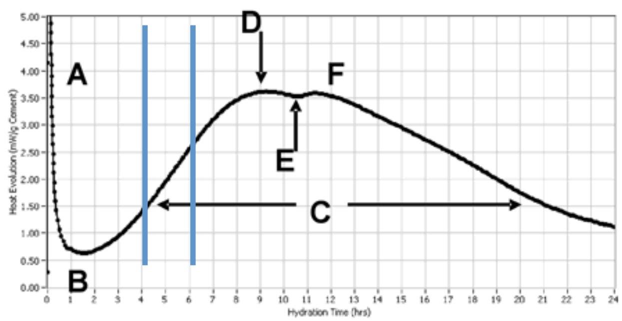

Figure 1, Early cement hydration captured by isothermal calorimetry at 23°C.

The typical setting time window for most concrete mixtures is indicated during the acceleration period between the end of the dormant period B and the maximum D. —(A) initial thermal power by dissolution of cement and initial cement hydration; (B) dormant period associated with very low thermal power indicating slow and well-controlled hydration: (C) main hydration peak predominantly associated with hydration reactions contributing to setting and early strength development, with maximum at (D); and (E) sulfate depletion point, followed by (F) accelerated calcium aluminate activity.

The binder content, and therefore the water to cement ratio and the cement to aggregates ratio, strongly affect where exactly setting occurs on the steep power curve after the end of the dormant period. In practice this means that set time prediction using calorimetry tends to work very well for normal concrete in the w/c range of 0.35-0.70, but less well for ultra high performance concrete and cement paste if the w/c is below 0.35, because in those cases setting occurs too close to the end of the dormant period to be easily and reliably detected by a thermal measurement. As a consequence, the use of isothermal calorimetry for set time prediction is less suitable for most standard cement paste, but it works remarkably well for set time prediction of most mortar and concrete mixtures. Hence, it is highly recommendable to use mortar or concrete samples when doing setting time estimation with an isothermal calorimeter.

The convenient rapid screening method

A popular, simple and yet reproducible calorimetry-based method for estimating ***relative*** set times involves simply comparing the time it takes to reach approximately half the maximum of the main hydration peak in the heat flow curve (Power curve), as in figure 2.

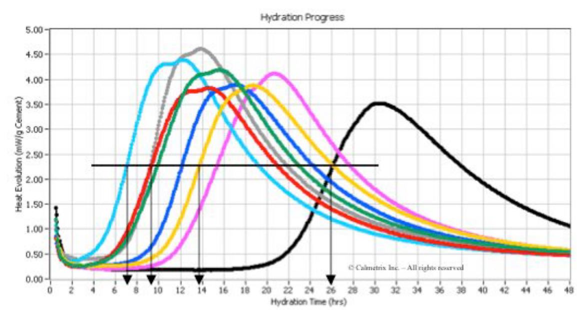

Figure 2. Estimation of relative set times using isothermal calorimetry heat flow curves.

The example in Figure 2 shows heat flow curves (Power curves) for mixes with different admixtures and different dosages. The relative position of the steep climb after the dormant period clearly indicates relative acceleration or retardation. For example, it can easily be inferred that the setting time for the mix corresponding to the yellow curve occurs approximately 4 hours after the setting time for the mix in the green curve.

A more quantitative approach

In practice the actual testing temperature in the calorimeter is likely to differ from that of a typical standard penetration set time test of mortar or concrete. Most standards, such as ASTM C403, use relatively large sample containers, which implies a fairly significant temperature rise in the order of several degrees C in the sample during the test. This leads to temperature induced variability and also a different temperature history if compared to the same mixture tested by isothermal calorimetry.

For a more precise estimation of settings times, one could first measure the average temperature and the penetration resistance of the concrete or mortar mixture of interest while doing traditional physical testing by penetration resistance. Then a calorimetry test can be done at the average temperature measured above, and penetration resistance values can be correlated to the exact point on the power curve where setting occurs. The Calmetrix I-Cal Set software module offers an easy way to correlate physical test data measured by penetration resistance to calorimetry curves.

The difference between the rapid screening method shown in Figure 2 and a more quantitative prediction is that the latter requires separate measurements to quantify the correlation between the physical results (i.e. penetration resistance) and the thermal response shown in Figure 2.

Note: Given the strong effect of water-cement ratio and cement content, as well as normal differences in reactivity between different binders, one should only use calorimetry for set time ***prediction*** of very similar mixtures in terms of cement binder type and amount, w/c ratio, and aggregates. For less accurate ***estimates*** of setting times, one may compare different binders and admixtures while keeping the general mix design nearly constant in terms of w/c, binder content and aggregates size and content.