calmetrix

Application Note AN-C03

Compressive strength prediction using isothermal calorimetry

Instruments to which this note applies: I-Cal 2000 HPC, I-Cal 4000 HPC, I-Cal 8000 HPC,
I-Cal 4000*, I-Cal 8000* (*: up to 48-hour strength estimate)

Target use: Mix design development with admixture, Quality Control at the Cement or Dry Mix plant

Introduction

Compressive strength testing of cement or dry mortar mixtures is labor intensive. It is also often difficult to do at a fixed water to binder ratio when using dispersants and other chemical admixtures, due to potential issues with bleeding and segregation. This application note describes how isothermal calorimetry can be used for a simple prediction of admixture effects on strength development for cement or dry mortar at a fixed basic mixture design (i.e. a fixed content of binder, water and sand), but with variable admixture content. This allows the user to predict and understand the effect of admixture on hydration kinetics and strength development with significantly less need for labor-intensive compressive strength data. The method described is useful for applications where one or a few binders are repeatedly used for admixture selection, formulation development or benchmark testing.

Figure 1 shows the heat flow curve from a hydrating fly ash blended cement in the absence of admixture, at a water-to-cement ratio of 0.45, tested at 23 °C in an isothermal conduction calorimeter. Figure 2 shows the corresponding integrated heat flow curve. By integrating the heat flow one gets the energy or heat of hydration, which is proportional to the degree of hydration of the cement.

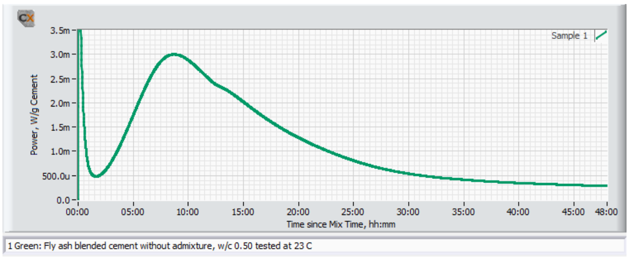

Figure 1. Heat flow curve for a fly ash blended cement normalized by weight of cement.

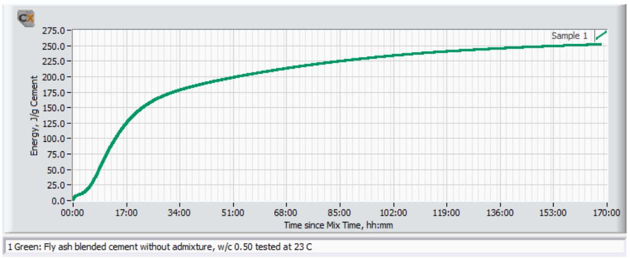

Figure 2. Heat of hydration (energy, integrated heat flow) for the fly ash blended cement in Figure 1, normalized by weight of cement.

Proven correlation between heat of hydration and compressive strength development

Figure 3 shows the actual correlation between heat of hydration and compressive strength for the fly ash blended cement in Figures 1-2, tested without admixture using a fixed mixture design. Heat of hydration (Energy) values beyond 7 days were extrapolated using Calmetrix' strength prediction software.

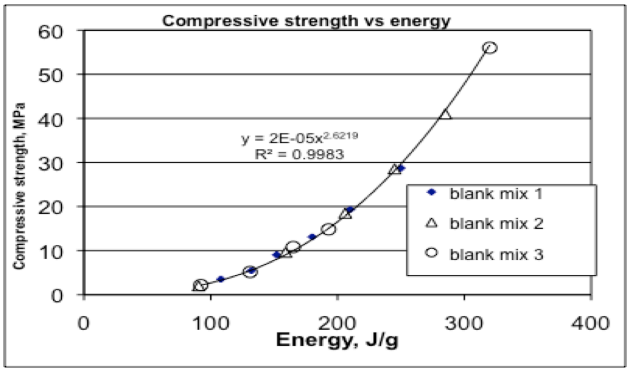

Figure 3. Correlation between energy / heat of hydration and compressive strength for cement mortar tested without admixture.

Figure 4 shows the heat flow curves obtained for mortar with the same cement and the same mixture proportions of cement, sand and water as in Figures 1-3, however with the addition of 4 different dosages of sodium gluconate. Figure 5 shows the corresponding heat of hydration curves. Figure 6 shows the compressive strength and heat of hydration for the mixtures with sodium gluconate added to the compressive strength – heat of hydration curve previously shown for the cement without admixture (Figure 3).

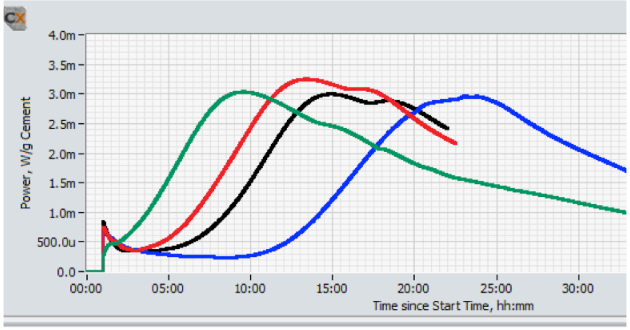

Figure 4. Heat flow curves for the same cement as in Figure 1 tested with 4 different dosages of sodium gluconate (0.02,

0.05, 0.08 and 0.15% sodium gluconate) normalized by weight of cement.

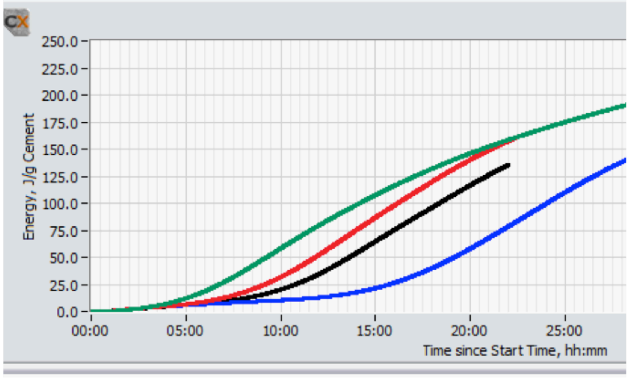

Figure 5. Heat of hydration (energy, integrated heat flow) for the same cement as in Figure 2 tested with 4 different dosages of sodium gluconate (0.02, 0.05, 0.08 and 0.15% sodium gluconate) normalized by weight of cement.

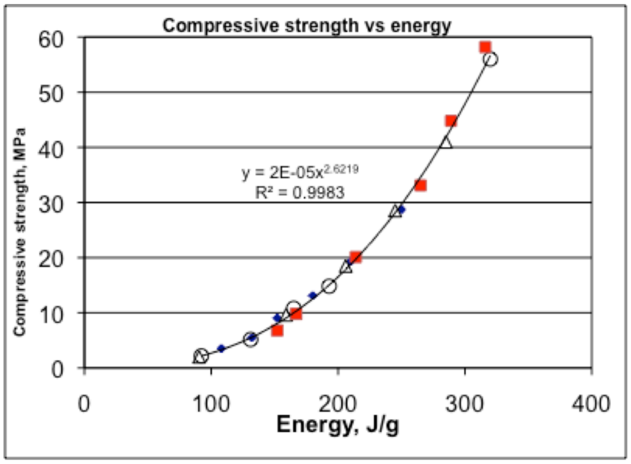

Figure 6. Correlation between energy / heat of hydration and compressive strength for cement mortar tested without admixture and with 4 different dosages of sodium gluconate. The red filled squares represents the mixtures with sodium gluconate.

Notes:

1) Sodium gluconate, a common retarder, significantly retards the cement hydration even at very low dosages, as seen in Figures 4-5. However, measured pairs of heat of hydration and compressive strength at four different dosages fit very well with the compressive strength – heat of hydration correlation for the cement tested without admixture, using the same mixture proportions except for the admixture dose, Figure 6.
2) The results imply that the effect of the admixture on cement hydration is primarily to alter the *rate* of cement hydration, not the hydration mechanism or the path of the cement hydration. As a consequence one can measure the admixture effect on cement hydration kinetics using isothermal calorimetry and use the results to predict the effect on compressive strength as long as the mixture design is constant except for the admixture dose.
3) By establishing the compressive strength – heat of hydration curve at different water to binder ratios, it is also possible to predict the effect of lowering the water content, as is often done when using dispersants.
4) For best results, the portion of the heat of hydration measured prior to initial set should not be used when establishing the correlation between energy and compressive strength development, and also not when predicting strength development from energy curves. In practice one can for each curve use the end of the dormant period after the initial peak, Figure 1, to establish when to start integrating the heat flow curve as a substitute for the labor intensive manual setting time method.

Test Protocol – strength prediction using isothermal calorimetry

The I-Cal Strength software helps users establish a model to correlate compressive strength with heat of hydration for a basic mix (i.e. a fixed content of binder, water and sand). This model can then be used for analysis and prediction of compressive strength of the same basic mix with varying types and dosages of admixtures. The test protocol is established as follows:

<u>Modeling:</u>

1. Set the calorimeter at the desired test temperature and wait for at least 16 hours for the calorimeter to stabilize.
2. Select a well known cement and a standard mixture design to be used for establishing the energy – compressive strength relationship.
3. Prepare one or several replicate mortar mixtures and measure the strength development and heat flow in parallel at given curing temperatures.
4. Measure compressive strength data at a minimum of four test ages ranging from a few hours after final set to 7 days. Typical test ages for a Portland cement are 12-16 h, 24 h, 48 h, 72 h, 120 h, 168 h. Make a note of the actual ages at time of testing within +/-10 minutes. Exclude any calorimetry data recorded prior to the end of the dormant period, at the onset of the main hydration peak (See Fig. 1) from the calculation of heat of hydration as a function of hydration time.
5. Input the collected energy and compressive strength data into the Modeling tool in I-Cal Strength to build a correlation between energy and compressive strength development that is specific to this cement and to this mixture design.

Compressive strength prediction:

Now that a model has been built for a basic mix design, users can follow the protocol below each time they test the same basic mix, with or without different admixtures and dosages.

6. Prepare one or several cement mortar mixtures using the same parameters as in step #3 and 6 except for the following modifications:
7. Include the admixture to be tested. If needed adjust the water to binder ratio within the range established in steps #3-6.
8. If using a dispersant, mix using the highest possible speed without causing splashing during mixing.
9. Carefully sample each cement mortar mixture prepared for calorimetry testing. If using a very fluid moisture, carefully stir the mortar mixture by hand while sampling into the calorimetry test vial to minimize any error in cement content caused by segregation.
10. Exclude any calorimetry data recorded prior to the onset of the main hydration peak (See Figure. 1) from the calculation of Energy / Heat of Hydration as a function of hydration time.
11. After completion of the heat of hydration test of cement mortar with admixture, use the correlation generated in the Model (steps 3-6), along with the measured heat of hydration for the cement mortar with admixture to estimate its strength development at different curing ages.

Conclusion

Isothermal calorimetry has the potential to substantially reduce the amount of compressive strength testing required when selecting admixtures or developing formulations involving one or a few standard binders. A similar and simplified approach (without admixture) can also be used at the cement plant level as a QC tool to predict early age strengths to aid the cement manufacturing process without the need for compressive strength testing.