

What Can We Learn From Monitoring Concrete Temperature?

by P. Taylor and J. Gajda

Synopsis: There is a growing interest in monitoring the temperature of cement paste, mortar and concrete, particularly at early ages. However, there also seems to be confusion about what is being achieved by this activity, and what to do with the information once it is recorded.

This paper outlines the tools and techniques in use, and discusses their applications, benefits and limitations. The discussion will cover concepts such as heat of hydration, maturity, isothermal calorimetry and semi-adiabatic temperature monitoring for assessing setting times, and potential incompatibility between the reactive ingredients (cements, supplementary cementitious materials, and chemical admixtures) in a mixture.

Keywords: heat of hydration; isothermal calorimetry; maturity; setting time; temperature

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INTRODUCTION

There is increasing interest and activity in using temperature monitoring techniques to measure different aspects of cement and concrete hydration. This is because cement develops heat while it hydrates, leading to an increase in temperature. Tracking this heat or temperature provides an effective means of monitoring the progress of the hydration. The heat generated also has implications for design of floors on grade, pavements and mass structures to accommodate the dimensional changes associated with rising and falling temperatures.

Along with the increased activity there is also increasing misunderstanding about the various techniques that are being used and their appropriate applications. This paper discusses the various techniques in use, including their benefits and limitations, and where and how they can be used. The topics covered include:

- Maturity
- Isothermal Calorimetry
- Heat of Hydration
- Setting Time Measurement
- Field Calorimetry

A critical aspect to be noted when considering all of these systems is whether the technique is isothermal, adiabatic or semi-adiabatic – or none of the above. Data collected under one condition may not be transferable or comparable to another condition.

Isothermal tests are controlled at a fixed temperature and often involve measurement of the energy required to maintain that temperature. These data are not representative of the temperature variations experienced in the field, but do provide useful, controlled information about relative hydration kinetics of the systems being investigated. Adiabatic tests are where the samples are sufficiently insulated, or controlled, such that there is no heat transfer to or from the environment. This is representative of concrete about 3 feet below the surface of large concrete elements at early ages, and may lead to very high temperatures. Semi-adiabatic systems are partially insulated, and the heat transfer to the environment is estimated or measured and allowed for. Such tests are

often simpler and cheaper to run because the insulation or control systems are much simpler. Other alternatives are simply to track the temperature in the structure, where the environment is not controlled.

MATURITY

The basis for this method is simple: the strength and modulus of elasticity of a concrete specimen are directly related to the quantity of heat developed from the hydrating cementitious materials. The technique is therefore to estimate the strength based on recorded temperatures of the in-place concrete and a model developed from laboratory testing of the same mixture¹.

Maturity methods can and have been used extensively to predict the in-place strength and strength gain of concrete in the field. The practical benefit of this method is that field evaluations of in-place strength can be predicted from simple measurements of the concrete temperature over time. This can lead to efficient usage of forms in structural concrete, and correct timing of saw-cutting in slabs on grade. The theoretical benefit is the ability to accurately predict both the strength and modulus of elasticity of concrete over a wide range of conditions based simply on the temperature development in the modeled concrete. A given maturity model, however, is only applicable to a single mix, and has to be re-calibrated if there are changes in the mix proportions or cementitious materials used. It should be noted that maturity testing does not indicate the quality of the concrete with respect to mix proportions, consolidation or surface finishing. The method also is based on the assumption that the concrete is kept moist, and is crack free.

Maturity testing entails developing a maturity curve (Figure 1) that correlates the development of particular concrete property for a specific concrete mix to both time and temperature. After the maturity curve is developed, the concrete property can be estimated from a measured time-temperature record of the concrete. The maturity function is a mathematical expression to account for the combined effects of time and temperature on the strength development of concrete. The key feature of a maturity function is the representation of how temperature affects the rate of strength development.

There are different approaches to developing the models needed to use maturity effectively. ASTM C 1074 provides procedures for using the measured in-place maturity index to estimate in-place strength (Figure 1). This practice describes two maturity functions. The first, and most popular for use with concrete pavements, is the Nurse-Saul time-temperature function. The other, preferred, maturity equation is the Arrhenius equivalent age function. This function presents maturity in terms of the equivalent age of curing at standard laboratory conditions. Although the equivalent age maturity function presents results in a more understandable format (the equivalent age), the complexity of its equation is likely why this method is less popular than the time-temperature factor method.

Equivalent age is expected to result in more accurate results when large temperature changes occur in the field. However, the time-temperature method is easier to apply and

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has been successfully used for estimating the in-place strength of paving concrete. Each of these functions requires preliminary testing to relate to the strength of concrete. Each method has a constant that can be assumed or for accuracy determined for the specific mix (ASTM C 1074). In the temperature time factor, a datum temperature below which hydration is minimal is assumed to be 0°C, or can be determined experimentally. In the equivalent age method, activation energy is assumed to be 33500 J/mol, or can be calculated.

ASTM C 918 uses the maturity method of monitoring temperature of cylinders cured in accordance with standard methods outlined in ASTM C 31 (AASHTO T 23). Cylinders are tested at early ages beyond 24 hours, and the concrete temperature history is used to compute the maturity index at the time of test. Using historic data, a prediction equation is developed to project the strength at later ages based on the maturity index and early-age strength tests².

Maturity can be determined with commercially available test equipment (Figure 2), or with standard temperature-logging equipment and an understanding of maturity. In either case, temperature sensors must be placed at critical locations in the concrete. For most pavement placements, this will be either at the top or bottom surfaces. When in doubt, use the lower of the maturities from sensors at the top and bottom surfaces. For mass and structural concrete, sensors are placed at the surface for maturity applications.

Commercially-available test equipment (maturity meters) log the concrete temperature as a function of time, and presents the current maturity (time temperature factor or equivalent age) of the concrete. There are two primary types of maturity meters. The first type uses temperature sensors in the concrete and has logging equipment outside the concrete. The second type combines the temperature sensor and logging equipment in a single package, which is embedded in the concrete. Wires extend from the sensor to outside the concrete. These wires must be periodically connected to a hand-held reader so that the maturity data from the sensor can be read. Wireless equipment is also available. Maturity can also be calculated from temperature sensors (such as thermocouples, thermometers, or thermistors) that are embedded in the concrete. The time interval should be selected to adequately resolve temperature changes in the concrete. In most cases, hourly time intervals are adequate.

ISOTHERMAL CALORIMETRY

In isothermal calorimetry testing, a small paste sample is maintained at a fixed temperature. The rate of heat evolution is monitored and plotted in units of specific power (rate of energy transfer per unit mass) versus time. The shape of the curve is descriptive of the hydration kinetics of the paste³. An example is shown in Figure 3 showing the retardation effects or increased water reducing admixture dosage.

By varying the materials in the paste and their proportions, the effects of supplementary cementitious materials, chemical admixtures, cement composition, and batching sequence

can be addressed. The technique is also useful for determining whether the system being tested contains sufficient sulfates, which influence time of set and early rate of hydration.

An isothermal heat conduction calorimeter consists of a heat sink that has two heat flow sensors and a sample holder attached to each sensor. A freshly prepared mixture is placed in a container in contact with one of the holders and a thermally inert material is placed in contact with the other. The heat released by the reacting cementitious sample passes across a sensor into the heat sink. The output from the calorimeter is the difference between the outputs from the sample heat flow sensor and the inert heat flow sensor. The measurement therefore takes place at essentially constant temperature.

The relative hydration performance of test mixtures are compared with appropriate control mixtures. The procedure and apparatus can be used to assess potential incompatibilities including:

- Effect of addition of sulfate on the setting time of a mixture. This can be used to indicate whether a cementitious system (including admixtures and supplementary cementitious materials) has the optimum amount of sulfate.
- Relation between the time of maximum alite heat evolution and the time of sulfate depletion. This relationship may be used as an indicator for the overall sulfate balance of each mixture.
- Acceleration of aluminate hydration before setting as a function of admixture type and dosage.
- Retardation of the alite hydration as a function of admixture type and dosage.
- Effect of supplementary cementitious materials type and dosage relative setting time at constant temperature.
- The rate of heat evolution from cementitious pastes as well as combinations of cements with chemical and mineral admixtures.

The equipment is relatively expensive and the data are sensitive to procedures in the laboratory. The method in preparation at ASTM calls for mixtures to be prepared outside the machine because precision is improved if mixing is complete and if it is uniform between mixtures. This approach does mean that the first readings are taken 5 to 10 minutes after water is added to the cement, thus losing some of the data associated with aluminate reactions. This is generally considered acceptable in order to gain better data about the silicate reactions. The initial peak is also associated with the heat of wetting the dry materials, and poor correlation has been found between this first peak and the physical performance of the cementitious system⁴. Some calorimeters allow internal mixing if the data are required for determination of heat of hydration (see following section) or other purposes.

HEAT OF HYDRATION

Reactions involving cementitious materials are exothermic, leading to a rise in temperature. The relative rates of heat generation provide useful information on the hydration kinetics of the systems as discussed above. Another effect of this temperature rise is that measures have to be taken to prevent cracking resulting from dimensional changes associated with the temperature changes. Knowledge of the heat generated by

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the system is a required input parameter in numerical models (ACI 207) used to predict the risk of thermally related cracking. Heat of hydration is expressed in units of energy per unit mass at fixed time intervals (e.g. kJ/kg at 7 days). Additionally, ASTM C 150 imposes optional maximum limits on the heat of hydration of Type II and IV cements, based on measurement of the heat of solution in accordance with ASTM C 186.

ASTM C 186 is not considered an ideal test because the precision is poor⁵, it is costly, not all laboratories have the required equipment and such standard equipment is not commercially available. Investigations are underway to develop alternative methods of measuring heat of hydration of cementitious materials. Such approaches include

- Isothermal conduction calorimetry (using the same calorimeter discussed above)
- Adiabatic calorimetry using mortar or concrete samples in an adiabatic chamber
- Semi-adiabatic calorimetry using simpler equipment and allowing for energy loss from the chamber.

All of these approaches have strong points and limitations, with the use of the isothermal conduction calorimeter reportedly showing most promise. This test is suitable for testing paste samples for a relatively short period because it does tie up a calorimeter for the length of the test. An advantage is that data collection is at frequent time intervals, leading to a smoother curve than in the incremental C 186 approach, where normally only two ages are used. Work is still required to better understand the precision and bias of the method, and to ensure that existing limits are still appropriate. Examples of comparative data for the same sample are shown in Figure 4.

SETTING TIME

One clear indication of when silicate reactions are starting (indicating setting) is a rapid increase in temperature at the end of the dormant period. Devices that measure the temperature of the in-situ concrete or laboratory samples allow the time of set to be determined at relatively low cost (Figure 5). Work is ongoing at ASTM to standardize such an approach including a round robin of a draft method conducted in 2005. Correlation with initial set is relatively simple, and is either taken at the point of inflection of a time-temperature plot, or the point when the second derivative of such a plot is at zero.

Attempts to determine the final set are less successful, with attention being focused on the slope of the first derivative of the time – temperature curve. This lack of correlation is not surprising because the current ASTM C 403 definition of final set is based on an arbitrary figure of pressure applied through a penetrometer rather than any parameter associated with the hydration kinetics of the system. The rate of hydration will vary between different systems and the time taken to achieve a given resistance to pressure may not be related to the slope of a time-temperature plot. Data has shown that final set occurs between the top of the time-temperature curve and about half way up the curve (Figure 6)⁴. For a given mix, correlation between initial set and final set was reasonable, therefore final set can be predicted based on the initial set value.

Tracking data using sensors embedded in a structure will allow better timing of activities such as saw-cutting joints in pavements and floors on grade. In the laboratory it is easy to automate data collection, therefore staff do not have to work a late shift if mixtures are made late in the day.

FIELD CALORIMETRY

There are many variations of field calorimeter in use, from simple thermocouples inserted in a concrete slab or cylinder, up to multi-celled semi-adiabatic systems with computer based data-loggers (Figure 7). The selection of the equipment will depend on the application and the data to be collected. Potential applications are those discussed above, and include:

- Collection of data for maturity monitoring
- Field assessment of potential incompatibilities between mix ingredients⁶
- Tracking uniformity of delivered materials using control charts of peak temperature and/or time of set
- Monitoring time of set for field batches

In general, the data collected from such systems is less precise than that from more expensive laboratory equipment, but it is easily transportable, and for many applications, the data are good enough to provide useful information about the performance of the system under investigation. Examples of such data are shown in Figure 8.

SUMMARY

A large amount of useful data can be collected by monitoring the temperatures of cementitious systems. These data are useful for characterizing the materials in the systems, investigating interactions between them, tracking uniformity, timing finishing activities that are governed by the time of set and monitoring strength development. The range of applications is wide, and the types of equipment available are varied. This paper has set out to clearly describe the different techniques that can be used, their applications, strengths and limitations.

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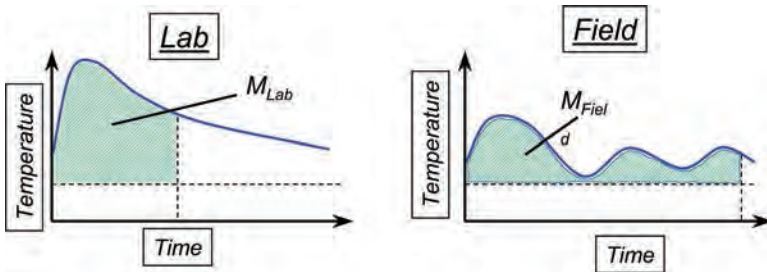


Figure 1—Maturity of the field concrete is equivalent to maturity of the laboratory concrete when the area under the curves is the same.



Figure 2—An example of a maturity logger.

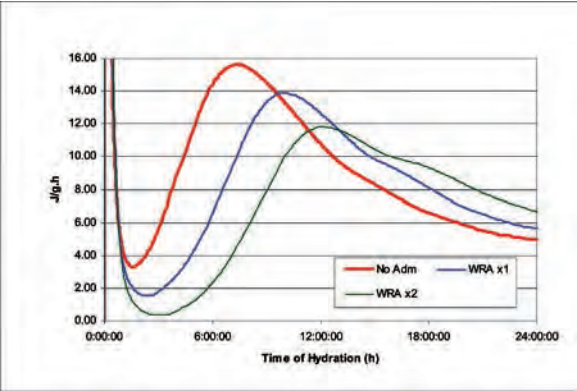


Figure 3—Example of isothermal calorimetry plots illustrating the retarding effect of increased dosage of a water reducing admixture. Highest temperature and earliest set is with the plain mixture, with lower peak and longer setting with increasing WRA dosage.

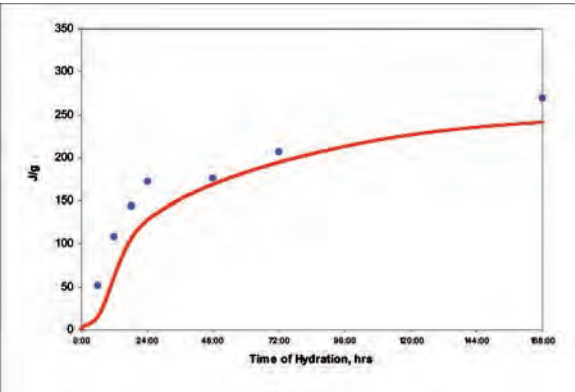


Figure 4—Example of data collected using ASTM C 186 heat of solution (dots) and an isothermal calorimeter (line) for the same cement sample.



Figure 5—Example of equipment used to measure setting time by monitoring temperature rise.

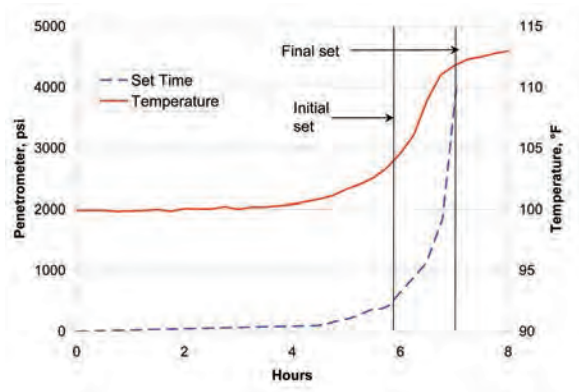


Figure 6—Data of semi-adiabatic temperature rise compared with penetrometer readings for the same concrete mixture (Taylor et al., 2006).



Figure 7—Field Calorimeter (Courtesy of Paul Sandberg, Grace).

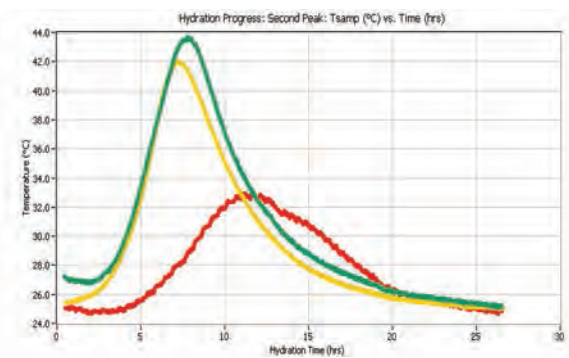


Figure 8—Typical data from a field calorimeter showing the effects of different cementitious systems (Courtesy of Gary Knight, Holcim).

Monitoring and Evaluation of Cement Hydration by Semi-Adiabatic Field Calorimetry

by J.P. Sandberg and S. Liberman

Synopsis: This paper describes the use of a recently developed, inexpensive portable semi-adiabatic calorimeter for monitoring cement hydration in concrete and mortar. The calorimeter measures the temperature as a function of time at the bottom of eight individual 3x6 cylinders with concrete or mortar. The measured temperature profile is used to evaluate the overall hydration performance of cementitious mixtures, with special emphasis on the timing and the size of the main hydration exotherms that strongly affect setting and early strength development of cementitious mixtures. Furthermore, a method has been developed for a more precise calculation of “thermal set”, with good correlation to manual set times according to ASTM C403. The field calorimeter is useful to screen the effect of type and dosage of admixtures and supplementary cementitious materials on “thermal” setting times in concrete and mortar.

Keywords: cement-admixture interactions; cement hydration; compatibility; semi-adiabatic calorimetry; setting

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Sergey Liberman received his MS in Applied Physics from Leningrad Polytechnic Institute, USSR. Between 1989 and 1996, he worked as a research physicist in several US companies. In 1996, he founded Solidus Integration, a company specializing in development of automated data acquisition systems and instruments. Since then, the company has developed several instruments and software packages used for characterization of concrete and mortar.

INTRODUCTION

Isothermal heat conduction calorimetry testing of cement paste has long been used for research on the effect of mineral and chemical additives on the hydration kinetics of portland cement¹⁻⁴. While isothermal calorimetry with proper temperature control offers outstanding accuracy and repeatability, the following drawbacks have limited its use in the field:

- High cost
- Small sample size required to obtain isothermal conditions, thereby making direct studies of concrete impractical.

More recently, inexpensive semi-adiabatic calorimeters have gained popularity because of significantly lower cost than isothermal calorimeters, easy field application, and ability to test larger samples such as concrete or mortar. It has been shown that simple temperature profile measurements in cementitious materials can be used to predict abnormal setting and poor strength development and investigate the causes thereof⁵.

Description of a simple semi-adiabatic field calorimeter

Figure 1 shows a simple, 8 cell semi-adiabatic field calorimeter designed for testing concrete, mortar or paste with no need to remove coarse aggregates. Standard 3x6" plastic cylinders with lids are used as sample vials. Thermal sensors are located immediately underneath and in direct contact with each cylinder. The insulating styrofoam body of the calorimeter is mounted with data loggers and sensors in a rigid traveling case on wheels for easy transport.

RESEARCH SIGNIFICANCE

Modern concrete technology frequently involves not only cement from multiple sources, but also multiple sources of supplementary cementitious materials (SCM) and multiple admixtures. Although the materials used by themselves generally perform well and comply with existing standards and specifications, incompatibility problems occur, typically resulting in variable set, poor strength development, difficult finishing and often rejected concrete. Semi-adiabatic calorimetry is an inexpensive, yet easy to use and powerful field technique for monitoring the hydration performance of cementitious mixtures. It is especially useful for identification of dosage limits for SCM and admixture during mixture design optimization, as well as for QC of concrete batches.

COMPATIBILITY TESTING

Semi-adiabatic calorimetry is well suited for testing the compatibility of cement in combination of SCM and admixture in paste, mortar and concrete. A severely delayed alite hydration peak leading to a long initial set time and low early strength gain indicates incompatibility among portland cement, SCMs and admixtures. Past experience²⁻⁵ indicated that a major source of incompatibility is a lack of soluble sulfate for proper control of aluminate hydration in mixtures with significant amount of SCM and admixture. Portland cements are optimized for sulfate balance using standard tests without SCM or admixtures. Problems however may occur at high levels of SCM's and/or with high admixture dosages, and especially at elevated curing temperature since the solubility of calcium is depressed at high temperature. Not only will the use of SCM "dilute" the amount of soluble calcium sulfate provided by the portland cement. Class C fly ash that often contains significant amounts of reactive aluminate without providing any soluble calcium sulfate will consume significant parts of the soluble calcium sulfate and therefore make the *mixture* potentially sulfate deficient and prone to severe retardation of alite hydration. Most dispersing admixtures in turn will, because of improved dispersion and wetting of the binder, further increase the reactivity of the aluminate in both cement and the class C fly ash, thereby further increase the need for soluble calcium sulfate *in the mixture*. Figure 2 shows temperature profiles recorded in a compatibility study of an ASTM Type I cement with 20% substitution for class C fly ash with two ASTM Type D water-reducing and retarding admixtures. The mixtures were prepared as C109 mortars with w/c 0.50 at room temperature. Note that the portland cement by itself was properly optimized and the incompatibility problem only occurred in mixtures with 20% class C fly ash and an overdose of chemical admixture, which was verified in concrete as well.

Discussion of compatibility testing results

The green curve shows the cement – 20% class C fly ash mixture tested without admixture. After some initial heat evolution (A) mainly due to dissolution and aluminate hydration, the main alite hydration peak starts after about 3 hours hydration (B). Setting occurs after about 5 hours (C) and the remainder of the broad alite peak (D) that follows is largely responsible for the early strength development.

The yellow curve shows the cement – 20% class C fly ash mixture tested with nominal dosage of admixture. Note the somewhat accelerated aluminate hydration during the first hour (E). The strength giving alite hydration is retarded and starts after about 10 hours (F) in this example, which is typical for the admixture type and dosages used. Note however that the overall size of the broad alite hydration peak (G) is reduced compared to the mixture without admixture (D), which could indicate that the mixture is close to being out of balance³, and may suffer from slower than expected strength development.

The red curve shows the cement – class C fly ash mixture tested with a 25% overdose of admixture. At this dosage the aluminate hydration is not properly controlled, as indicated by the sudden and very sharp exotherm starting at about 2 ½ hrs hydration (H) as compared to the slope of the other two curves. The excessive retardation was confirmed by manual setting time in concrete. Note the sudden temperature increase, which we find

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typical for uncontrolled aluminate hydration³, compared to the slower and more gradual temperature rise caused by the strength giving alite hydration (D and G). In this case, the uncontrolled aluminate hydration in the overdosed mixture severely retards the onset of the alite hydration peak³, to the extent that no alite hydration can be seen even after 17 hrs of hydration (J). If in doubt, the aluminate and alite hydration can be measured by XRD on parallel samples to verify which phases are generating the exotherms.

Experimental designs for compatibility testing

It is very important to test for the effect of overdosing admixtures as well as supplementary materials in any compatibility study. Most if not all admixtures and SCM's can cause compatibility problems if overdosed, but often no signs of problems can be seen until dosages are close to the limit at which abnormal hydration occurs. Even when a mixture is close to abnormal hydration, it may appear normal until a minor change often very abruptly pushes the mixture beyond its limits into abnormal hydration. The "nominal dose" mixture (yellow) in Figure 1 would probably not be considered potentially problematic if the overdose test had not been performed. The overdose test in this case indicated that even a relatively small increase in admixture dosage (+25%) would cause the mixture to hydrate abnormally (red). Experience has shown that most field concrete mixtures need larger margins of safety to cope with normal changes in materials, temperature, etc. In our opinion, mixtures should be able to accommodate more than a 50% increase in admixture dosage without causing abnormal hydration. Table 1 shows a typical 8-mixture experimental design for basic compatibility studies. Further tests may include the effect of curing temperature, admixture dosing sequence, as well as alternate admixtures and SCM's. Concrete tests are recommended to confirm any key features obtained by testing of mortar or even paste.

The calcium sulfate addition test

Since many incompatibility problems are related to a too low availability of soluble calcium sulfate in the mixture with SCM and admixture, it is highly recommended to test for the effect of adding gypsum or plaster to the cement before testing whenever incompatibility issues have been detected. Although it may not be possible to add more soluble calcium sulfate to the cement during commercial production, the results of this test may provide important knowledge on the cause of an incompatibility problem.

Figure 3 shows the results of the sulfate addition test applied to the overdosed mixture (red) in Figure 2. Note how with the extra calcium sulfate the overdosed mixture hydrates normally as compared to the same mixture without added sulfate in Figure 2. With the added soluble calcium sulfate, the undesired peak related to uncontrolled aluminate hydration at ~ 3 hrs is gone (H), indicative of the extra calcium sulfate doing its job to control the aluminate³. As a consequence, a strong alite hydration peak now appears with a maximum at ~ 17 hrs (K).

PREDICTION OF SETTING TIMES

Recent work has explored the use of semi-adiabatic calorimetry to generate "thermal" setting times and their correlation with manual setting times according to ASTM C403.

Figure 4 shows the measured repeatability of 8 replicate concrete mixtures without admixture tested in the semi-adiabatic calorimeter seen in Figure 1.

Two alternate methods, the “Derivatives” method and the “Fractions” method, are considered for estimating “thermal” setting times from semi-adiabatic temperature profiles. The “Derivatives” method defines the time of the maximum slope (maximum first derivative) of the main alite hydration peak as final set, and the time of maximum 2nd derivative as initial set, Figure 5 (left). The “Fractions” method defines a baseline and a maximum temperature, and then calculate initial and final set as the time when the measured temperature has risen a certain percentage from the baseline towards the maximum temperature measured, Figure 5 (right). Figures 6-8 show the correlation for the “Derivatives” method and the “Fractions” method respectively, for concrete tested against manual setting times measured on the same mixture following ASTM C 403. Note that the data presented represents a compilation of multiple ASTM Type I and II cement sources and class F fly ash sources and contents 0-20% cement replacement, with water-cement ratios in the range 0.5-0.7. The concrete contains various compositions and dosages of ASTM Type D water reducing and retarding admixture.

The “Fractions” method offers the possibility to develop more accurate “refined” fractions for a given mixture design by comparing it against ASTM C403 manual set. Figure 8 shows the corresponding correlation for the “Fractions” method when it was refined for each cement type and w/c used. For the range of concrete mixtures tested, the fractions with best fit varied from 19-32% for “Initial thermal set” and from 42-64% for Final thermal set”.

Discussion on thermal setting times

The measured temperature-time curves for estimating initial and final set are semi-adiabatic and thus heat-loss and equipment dependent. Further work is required to establish the possible use at different temperatures, which was not possible in this work as only one curing temperature (room temperature) was used. The purpose of this work is merely to demonstrate the possibility of using inexpensive semi-adiabatic calorimeters to predict setting times as measured manually according to ASTM C403.

SUMMARY AND CONCLUSIONS

Semi-Adiabatic calorimetry has shown itself to be an inexpensive yet useful tool for compatibility studies of different materials such as portland cement, SCM’s and admixtures. Semi-adiabatic calorimetry is less accurate than isothermal calorimetry, but offers the advantage of being able to test concrete directly without the need to remove coarse aggregates. The laboratory data presented indicate that semi-adiabatic temperature curves can also be used to derive “Thermal” Setting times that correlate well with manual setting times measured according to ASTM C403. However, further work is needed to develop and verify a “thermal” method that works at different and perhaps variable curing regimes.

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Table 1 -- Basic experimental design for compatibility studies including one admixture and a class C fly ash.

Mixture	Dosages		Remarks
#	SCM	Admixture	
1	20% class C fly ash	None	Reference
2	20% class C fly ash	Low	Admixture dosage window of interest
3	20% class C fly ash	Typical	
4	20% class C fly ash	High	
5	10% class C fly ash	Low	Fly ash and admixture dosage window of interest
6	10% class C fly ash	High	
7	30% class C fly ash	Low	
8	30% class C fly ash	High	

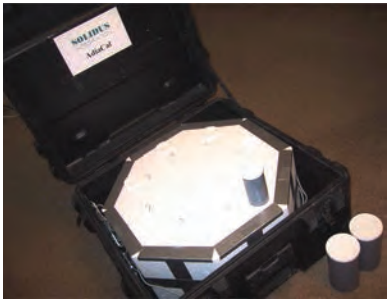


Figure 1 —AdiaCal semi-adiabatic field calorimeter.

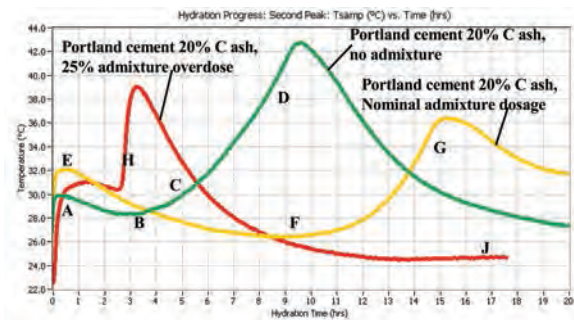


Figure 2—Compatibility study of an ASTM Type I cement with 20% substitution for Class C fly ash and a Type D water-reducing and retarding admixture.

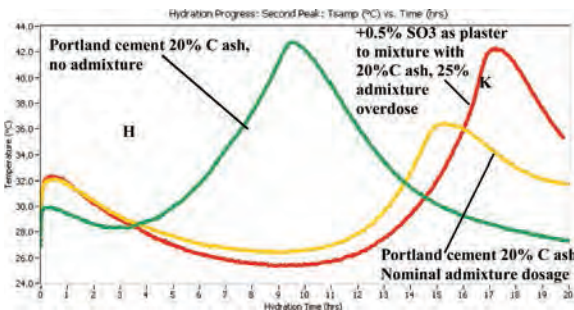


Figure 3—Effect of adding calcium sulfate to the abnormally hydrating mixture with overdosed admixture.

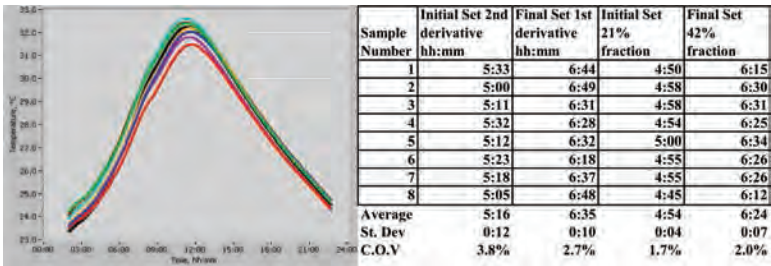


Figure 4—Measured repeatability of eight replicate concrete mixtures.

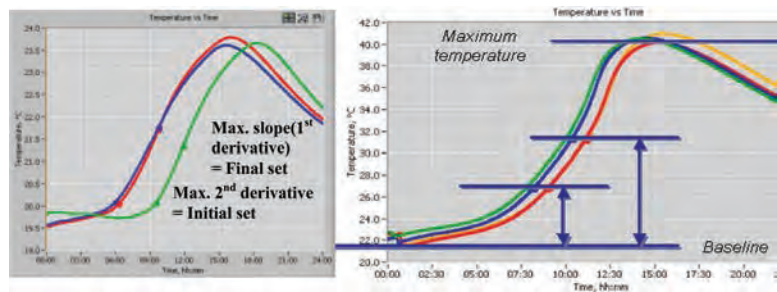


Figure 5—Calculation of “thermal set” using the “derivatives method” (left) and the “fractions method” (right).

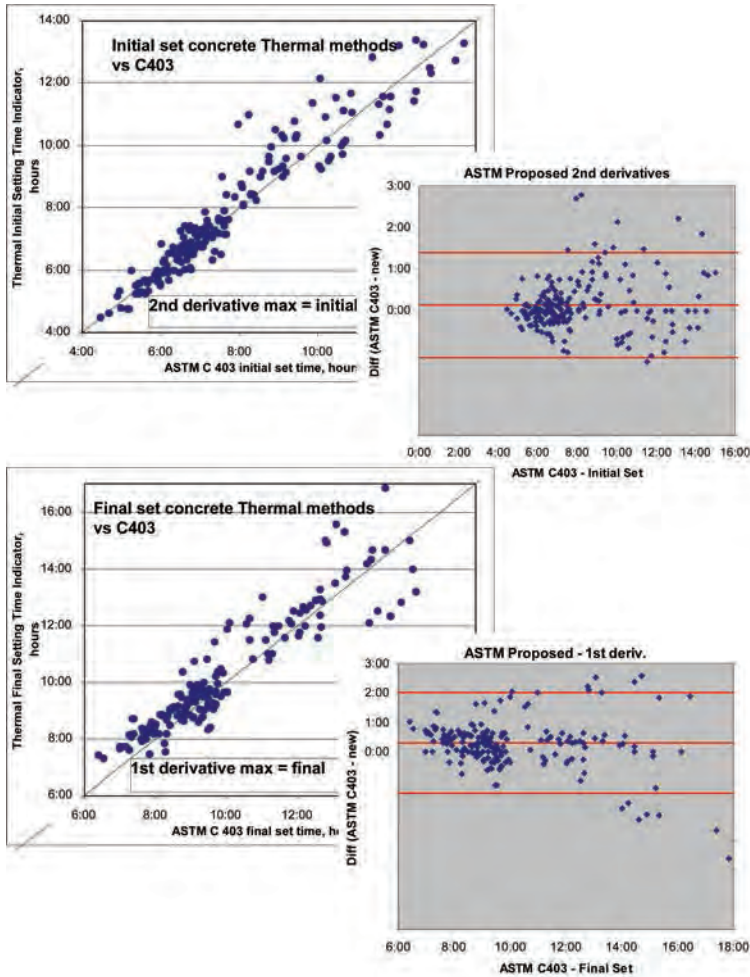


Figure 6—Correlation between “thermal” set using the “derivatives” method and ASTM C 403 manual set. (Top: initial set, bottom: final set. The right graphs show the 95% confidence interval limits. The standard deviation for eight replicate mixtures for the “derivatives” method was 38 minutes for “initial thermal” set and 52 minutes for “final thermal” set.)

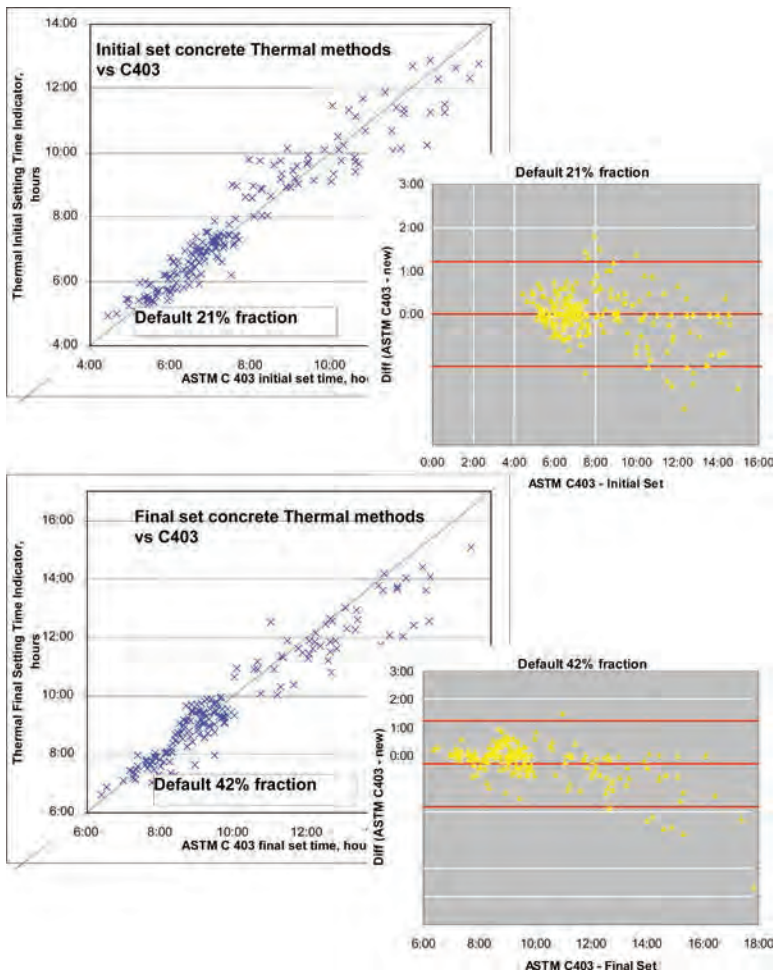


Figure 7—Correlation between “thermal” set using the “fractions” method and ASTM C 403 manual set. (Top: initial set, bottom: final set. The right graphs show the 95% confidence interval limits. The standard deviation for eight replicate mixtures for the “fractions” method was 36 minutes for “initial thermal” set and 46 minutes for “final thermal” set.)

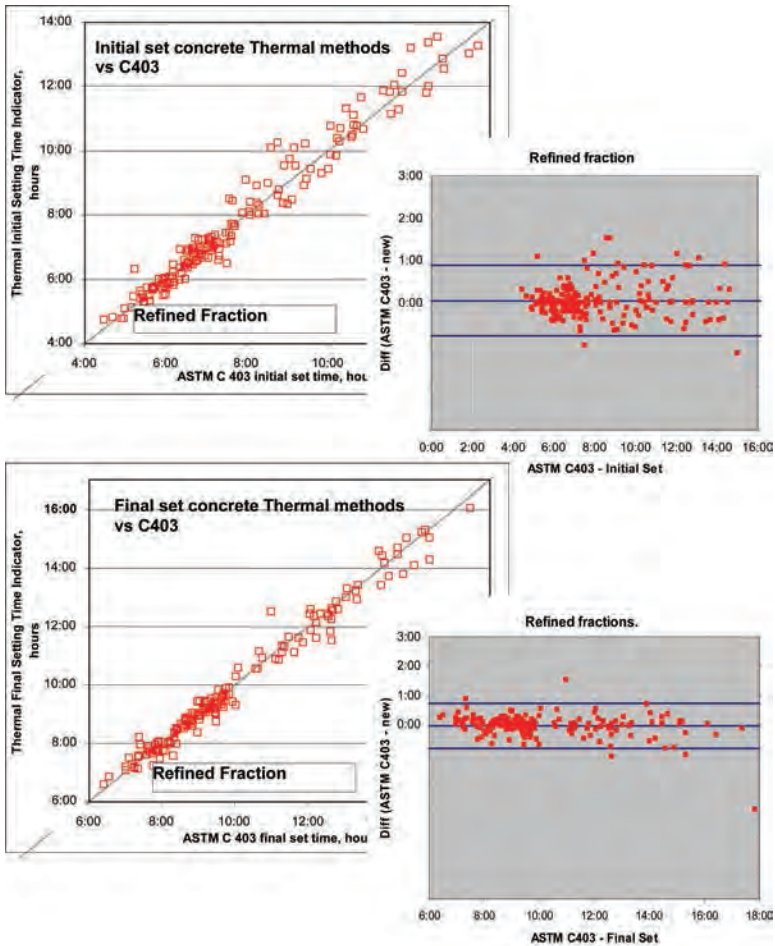


Figure 8—Correlation between “thermal” set using the “refined fractions” method and ASTM C 403 manual set. (Top: initial set, bottom: final set. The right graphs show the 95% confidence interval limits. The standard deviation for eight replicate mixtures for the “derivatives” method was 25 minutes for “initial thermal” set and 46 minutes for “final thermal” set.)

Calorimetry Performed On-Site: Methods and Uses

by K.A. Riding, J.L. Poole, M.C.G. Juenger, A.K. Schindler,
and K.J. Folliard

Synopsis: An accurate and practical method of determining the heat development of concrete mixtures under real mixing, cooling, hauling, placement, and curing conditions would greatly benefit contractors and engineers in helping predict in-place concrete member temperatures. Semi-adiabatic calorimetry was performed at several construction sites in temperature controlled rooms using concrete sampled from concrete placements. Semi-adiabatic calorimetry was also performed for comparison with concrete made under laboratory conditions from materials sampled at the respective batch plants. An energy balance-based finite difference method is presented for calculating the concrete non-linear heat generation using the measured heat of hydration determined from semi-adiabatic calorimetry. This method was used in a program which allows the direct input of values from semi-adiabatic calorimetry testing and estimates the development of in-place temperatures in mass concrete members of various geometries. Estimated concrete member temperatures are compared to the values measured on-site. Best practice suggestions are also given for performing semi-adiabatic calorimetry using concrete sampled on-site.

Keywords: calorimetry; concrete heat development; temperature prediction

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INTRODUCTION

Concrete temperature control has become a major issue as concrete bridge member sizes have increased. The cement content of the mixtures in these elements is often quite high in order to meet strength requirements. Also, the placement temperatures of such mixtures have often not been controlled. Finally, aggregate size is often low to meet placement requirements. These issues can lead to an increased risk of thermal cracking, and hence poor long term durability. To deal with these problems, engineers and contractors need a reliable, cost effective, and simple method to assess the thermal behavior of the concrete mixture under job-site conditions. An improved knowledge of the material behavior in-situ could help practitioners improve overall structural durability, cost, and construction sequencing.

Concrete mixing, hauling, and placing conditions can vary widely depending on the location and circumstances. Most concrete heat of hydration tests are performed in laboratories from concrete made using laboratory mixers. Ready mixed concrete can experience different amounts of agitation than laboratory made samples. The project location, such as over a large body of water, can also effect the conditions the concrete experiences before placement. Little work has been done on determining the effects of different mixing and hauling conditions on the temperature rise of concrete.

This paper presents the results of tests comparing the heat of hydration of concrete sampled at five different construction projects and performed onsite to replicate laboratory concrete mixtures performed under more controlled conditions. Semi-adiabatic calorimetry results were used to calculate the concrete adiabatic temperature rise in both cases. Also presented is an energy balance approach for determining the

temperature rise of a concrete member using the heat of hydration results obtained from semi-adiabatic calorimetry. The concrete member temperatures were predicted using a finite difference control volume based concrete temperature prediction program. Actual concrete member temperatures results are compared to the predicted values.

RESEARCH SIGNIFICANCE

Concrete temperature control is becoming increasingly important in concrete bridge construction where these issues have not been traditionally considered. Engineers and contractors need a reliable way of testing the concrete heat of hydration under job-site conditions, as well as a way of quickly predicting the concrete member performance using that concrete mixture. The work presented here is aimed at simplifying the estimation of concrete temperature by investigating the influence of mixing and testing location on test results and demonstrating the use of a free, user-friendly software package for temperature prediction.

EXPERIMENTAL METHOD

Concrete from five different construction projects in the state of Texas was tested onsite to determine their heat of hydration. The concrete placement location, concrete member type, and construction dates are shown in Table 1. Table 2 shows the mixture proportions used for each project. In some cases, tests were performed more than once onsite or in the laboratory.

The concrete heat of hydration was tested using a semi-adiabatic calorimeter. The semi-adiabatic calorimeter was placed in the nearest temperature controlled building or shed provided by the contractor. The concrete cylinders were made onsite using concrete sampled from the ready mixed truck or pump, and then transported to the nearby calorimeter. The concrete on the Galveston Causeway project differed from the rest because it was mixed and placed using a floating batch plant.

The semi-adiabatic calorimeter is an insulated steel drum with a cavity inside for a concrete cylinder. The semi-adiabatic calorimetry was performed on a 150mm by 300mm (6 in. by 12 in.) concrete cylinder. The temperature of the concrete and heat flux in the drum was recorded. The adiabatic temperature rise in the concrete was calculated from the concrete temperature and heat flux using the procedures suggested by RILEM Technical Committee 119¹ and a calibration procedure outlined in Poole et al.² An adiabatic temperature development curve is fit to the semi-adiabatic results using Equation 1^{3,4}:

$$Q_h(t) = H_u * C_c * \left(\frac{\tau}{t_e}\right)^\beta * \left(\frac{\beta}{t_e}\right) * \alpha_u * \exp\left(-\left[\frac{\tau}{t_e}\right]^\beta\right) * \exp\left(\frac{E}{R}\left(\frac{1}{273+T_r} - \frac{1}{273+T_c}\right)\right) \quad (1)$$

where Q_h is the rate of heat generation (J/hr/m³), H_u is the total amount of heat generated at 100% hydration (J/kg), C_c is the total amount of cementitious materials (kg/m³), τ is the hydration time parameter (hrs), t_e is the concrete equivalent age at the reference temperature (hrs), β is the hydration slope parameter, α_u is the ultimate degree of hydration (unitless), E is the apparent activation energy (J/mol), R is the universal gas constant (J/mol/K), T_r is the reference temperature (°C), and T_c is the concrete

temperature ($^{\circ}\text{C}$). H_u is calculated from the cement chemistry and supplementary cementing materials percentage.³ The activation energy is based on isothermal calorimetry performed on the cement, supplementary cementing materials, and chemical admixtures sampled onsite.⁵ The activation energies used in the calculations for M3 and M4 were based on isothermal calorimetry conducted on similar materials, because identical materials were not available.

Concrete constituent materials were sampled at the batch plant as close as possible to the concrete placement. In most cases, this was the same day as the placement. In the case of the El Paso project, the cementitious materials were sampled the week before concrete placement. Semi-adiabatic calorimetry was performed at the laboratory using the raw materials collected and the mixture proportions used on-site.

CALORIMETRY RESULTS

Results were obtained from semi-adiabatic calorimetry performed on-site and at the laboratory. The concrete heat of hydration parameters calculated from the semi-adiabatic calorimetry tests are shown in Table 3. Figures 1-5 shows the results from M1 through M5. There are some results that deserve further explanation in Figures 1-5. The materials used in test M1-L3 were sampled close to the placement of M1-1, and the results, correspondingly, match very well. The semi-adiabatic calorimeter used for concrete mixtures M3-1 and M5-1 was placed in rooms with rather poor temperature control. The room temperature in the job trailer used to store the semi-adiabatic calorimeter for M3-1 was very hot, and caused the heat flux from the concrete to fluctuate dramatically as shown in Figure 6. With test M5-1, the room temperature spiked at about 28 hours after mixing, as shown in Figure 7, due to a power outage or air conditioning malfunction. The measured heat flux correspondingly dropped. The change in heat flux in the M3-1 and M5-1 caused a change in the calculated hydration parameters, but not necessarily a large temperature difference between the field test and the laboratory test.

Table 4 shows the difference between adiabatic temperature rise parameters and the maximum difference in adiabatic temperature rise development for different tests. A difference of 8% for the α_u parameter, 17% for the τ parameter, and 15% for the β parameter can be considered significant differences between tests.² All of the tests compared, except M4, had at least one parameter with a difference between parameters great enough to be considered significant. The only tests that had a large difference in the adiabatic temperature rise development were M1-1 compared to M1-2 and M5-1 compared to M5-L2. This indicates that the adiabatic temperature rise curves were still quite similar. M1-1 and M1-2 were tested about 6 weeks apart from concrete sampled on-site. Because mixtures M1-1 and M1-2 were placed on over 6 weeks apart, a change in the cementitious materials between the two tests may have been the cause for the difference in these tests. Mixtures M5-1 and M5-L2 have a large difference in temperature because due to a batching error, M5-L2 contained less than half of the low-range-water reducing admixture as M5-1. This large difference in water reducer decreased the dispersion of the cementing materials, decreasing the degree of hydration.² The mixture was then repeated, resulting in a closer adiabatic temperature rise. This

indicates that semi-adiabatic calorimetry can be used as a quality control measure to detect large changes in materials or batch weights. It is recommended that the difference in hydration parameters and the difference between adiabatic temperature rise be examined for quality control testing purposes.

HYDRATION PARAMETER USE IN MODELING

The hydration parameters calculated as part of semi-adiabatic calorimetry can be used as direct inputs in an energy balance-based heat transfer analysis. Although temperature prediction of concrete members has been studied for some time^{6,7}, the heat transfer method used in this paper is presented for clarity and for reference as an easily implemented algorithm for calculating concrete temperature development. Equation 2 shows the heat diffusion equation:⁸

$$\frac{d}{dx}\left(k \cdot \frac{dT}{dx}\right) + \frac{d}{dy}\left(k \cdot \frac{dT}{dy}\right) + Q_h = \rho \cdot C_p \cdot \frac{dT}{dt} \quad (2)$$

where Q_h is the rate of heat generation (J/hr/m³), ρ is the density (kg/m³), C_p is the specific heat (J/kg/°C), and T is the temperature (°C). The concrete member may be idealized as a collection of control volumes, (or areas in the 1 or 2 dimensional case).⁹ Each control volume is assumed to have different, but uniform temperature and material properties. The change in temperature over a time step can then be calculated using an energy balance, as shown in Equation 3 and Figure 8:⁸

$$\dot{E}_{in} + \dot{E}_g - \dot{E}_{out} = \dot{E}_{st} \quad (3)$$

where $\dot{E}_{in}$ is the rate of energy entering the control volume (W/m³), $\dot{E}_g$ is the heat generation rate (W/m³), $\dot{E}_{out}$ is the rate of energy leaving the control volume (W/m³) and $\dot{E}_{st}$ is the rate of energy being stored in the control volume. The temperature change in the control volume can then be calculated using Equation 4:⁸

$$\Delta T = \frac{\dot{E}_{st} \Delta t}{\rho c_p dx dy dz} \quad (4)$$

where ΔT is the change in temperature in the control volume (°C), Δt is the time step used in the calculation (s), dx , dy and dz are the dimensions of the control volume (m).

The heat generated may be calculated using Equation 1. The incremental amount of heat generated during a time step used in the heat transfer analysis may be calculated as the total heat generated up to that time step (using Equation 1) minus the total heat generated from the previous time step. The energy entering the control volume from conduction (in 1 dimension) to the neighbor volume may be calculated using Equation 5, while the energy leaving the control volume from conduction may be calculated using Equation 6:

$$\dot{E}_m = \frac{dy_i \cdot T_{i-1}}{\frac{dx_{i-1}}{2 \cdot k_{i-1}} + \frac{dx_i}{2 \cdot k_i}} + \frac{dy_i \cdot T_{i+1}}{\frac{dx_i}{2 \cdot k_i} + \frac{dx_{i+1}}{2 \cdot k_{i+1}}} \quad (5)$$

$$\dot{E}_{out} = \frac{dy_i \cdot T_i}{\frac{dx_{i-1}}{2 \cdot k_{i-1}} + \frac{dx_i}{2 \cdot k_i}} + \frac{dy_i \cdot T_i}{\frac{dx_i}{2 \cdot k_i} + \frac{dx_{i+1}}{2 \cdot k_{i+1}}} \quad (6)$$

where k is the thermal conductivity for the control volume (W/m/°C), and the subscript refers to the representative control volume as shown in Figure 8.⁹ In the case of exterior boundary conditions that experience a radiation heat flux or convective energy exchange, the heat flux entering or leaving may be substituted for that found in Equations 5 and 6. The temperature distribution may then be solved using an explicit (forward) or implicit (backward) scheme. An implicit scheme will involve the simultaneous solution of the temperature values for all control volumes. In the case of the explicit scheme, a stability criterion such as the one for uniform thermal conductivity and control volume spacing shown in Equation 7 must be met to avoid divergence of the solution, which may require a small time step.⁹

$$\Delta t = \frac{\rho c_p (dx)^2}{2k} \quad (7)$$

In the explicit case, the temperatures values used for the calculations on the left hand side of Equation 3 are taken as the temperatures calculated during the last time step. The temperature calculated using Equation 4 is the temperature for the new time step.

During the early stages of hydration, the concrete material properties and heat generation values are changing rapidly. The concrete material properties, such as the thermal conductivity and specific heat are dependent on the temperature and degree of hydration. If the implicit method is used, the solution becomes very non-linear and requires iteration. The explicit case, however, does not have this limitation and requires no iteration. When using the explicit method, the thermal properties, boundary condition heat fluxes, and degree of hydration for the present time step may be calculated using the temperatures from the previous time step. Figure 9 shows a flow chart of the order in which the calculations are made during a time step. As seen in the flow chart, the concrete temperature may be calculated by updating the boundary conditions, material thermal properties, and degree of hydration by direct calculations (without any of the iterations required by other methods).

TEMPERATURE PREDICTION SOFTWARE

The procedure for calculating the temperature distribution in concrete outlined in this paper was used in a temperature prediction software package developed as part of this project. This software program can be used to quickly predict the temperature distribution for mass concrete members. When the concrete materials and mixture proportions used in the analysis do not meet the construction specifications, the inputs

may be quickly adjusted and the temperatures recalculated. This software provides users the ability to rapidly perform the “what if” scenarios to help lower cost and produce more durable concrete. The software provides a summary and graph of the maximum temperature and maximum temperature difference in the member with time. Also, the software will export the temperatures calculated at the control volumes with time to a text file or spreadsheet software.

Figures 10 and 11 show the measured versus predicted core and near surface temperatures for a footing placed using the concrete from M3-1 (onsite) and M3-L2 (laboratory). The measured temperatures compare quite favorably to those predicted using the hydration parameters measured for M3-1 and M3-L2 despite a difference of over 46% in the τ parameter, demonstrating that the hydration parameters measured onsite or in the laboratory may be used as direct inputs into temperature prediction software. The results shown are representative of those obtained for all mixtures, with only one being shown to conserve space.

It should be noted that temperature predictions made using adiabatic temperature development curves should be made using the most recent data available to minimize this difference. This is clear from the variations seen in the adiabatic temperature rise curves for mixtures M1-1 and M1-2, Figure 1, which were done about 6 weeks apart. Using the data from mixture M1-1 (or M1-L3) for concrete placed using the materials from M1-2 could give inaccurate results due to differences in the materials used with time.

CONCLUSIONS AND RECOMMENDATIONS

The temperature rise of concrete from five different construction projects was measured onsite and in the laboratory using semi-adiabatic calorimetry. The mixtures that were sampled onsite compared well to the mixtures replicated in the laboratory. This indicates that either practice is acceptable. It is recommended to test the temperature control of a room that will be used to store a semi-adiabatic calorimeter before testing, to minimize any resulting errors. As differences in the calculated hydration parameters may still give similar adiabatic temperature rise curves and consequently predicted temperatures, it is recommended that the difference in hydration parameters and the adiabatic temperature rise curve be used together to determine any differences in the material hydration behavior.

A method for easily calculating the concrete hydration using a spreadsheet or software package has also been presented. Adiabatic temperature rise parameters obtained through semi-adiabatic calorimetry may be used as direct inputs in temperature prediction calculations using the methods outlined in this paper.

ACKNOWLEDGMENTS

The authors wish to express their gratitude to the Texas Department of Transportation for funding this work and other on-going research. The advice and support of Ralph Browne, Tom Yarbrough, and Rob Crowson are greatly appreciated

NOTATIONS

GGBFS	=	Ground Granulated Blast Furnace Slag
F	=	ASTM C 618 Class F Fly Ash ¹⁰
C	=	ASTM C 618 Class C Fly Ash ¹⁰
MRWR	=	Mid Range Water Reducer
LRWR	=	ASTM C 494 Type A Low Range Water Reducer ¹¹
HRWR	=	ASTM C 494 Type F High Range Water Reducer ¹¹
SRG	=	Siliceous River Gravel
SNS	=	Siliceous Natural Sand
CG	=	Crushed Granite
CL	=	Crushed Limestone

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Table 1 – Concrete member construction information

Concrete Member Description	Member Size Length x Width x Height (m)	Mix ID	Construction Date	Location
Column & Footing	1.8 x 3.1 x 9.1	M1-1	6/16/2003	Round Rock, Texas
	3.1 x 3.1 x 1.9	M1-2	8/1/2003	Round Rock, Texas
	-	M1-L3	3/24/2004	Laboratory
Dolphin (a large concrete member used as a bumper to protect the causeway against barge impact)	4.9 x 4.9 x 2.7	M2-1	2/5/2004	South Padre Island, Texas
	4.9 x 4.9 x 2.7	M2-2	9/10/2004	South Padre Island, Texas
	-	M2-L3	3/24/2004	Laboratory
	-	M2-L4	5/10/06	Laboratory
	18.3 x 4.1 x 2.0	M3-1	8/9/2004	Galveston, Texas
Causeway Footing	-	M3-L2	11/29/2005	Laboratory
	1.0 x 1.0	M4-1	3/31/2004	Wichita Falls, Texas
Rectangular Bent Cap	-	M4-L2	5/5/2004	Laboratory
	2.7 x 1.8 x 1.7	M5-1	2/22/2005	El Paso, Texas
Pilaster		M5-L2	11/15/2005	Laboratory
		M5-L3	5/10/2006	Laboratory

Table 2 – Concrete mixture proportions

Member	M1	M2-1 & M2-L3	M2-2 & M2-L4	M3	M4	M5
Cement kg/m ³ (pcy)	253 (426)	253 (426)	244 (411)	167 (282)	251 (423)	181 (305)
SCM kg/m ³ (pcy)	64 (107)	100 (168)	109 (189)	133 (224)	64 (107)	165 (278)
Water kg/m ³ (pcy)	135 (228)	123 (207)	123 (207)	104 (175)	126 (212)	149 (250)
Coarse Aggregate kg/m ³ (pcy)	1041 (1754)	1112 (1874)	1084 (1827)	1109 (1869)	1108 (1867)	997 (1680)
Fine Aggregate kg/m ³ (pcy)	845 (1424)	687 (1157)	670 (1129)	793 (1337)	727 (1225)	746 (1258)
SCM Type	F	F	F	F	C	GGBFS
Coarse Aggregate Type	CL	SRG	SRG	SRG	CG	SRG
Fine Aggregate Type	SNS	SNS	SNS	SNS	CG	SRG
Chemical Admixtures	LRWR	LRWR MRWR	LRWR MRWR	LRWR HRWR	LRWR	MRWR

Table 3 – Concrete hydration parameters determined by semi-adiabatic calorimetry

Mix ID	H_u (J/g)	E (J/mol)	α_u	τ (hrs)	β
M1-1	465,210	29,410	0.654	14.76	0.860
M1-2	465,290	29,410	0.715	15.34	0.823
M1-L3	465,210	29,410	0.698	19.64	0.776
M2-1	455,720	21,150	0.760	12.74	0.952
M2-2	451,620	21,150	0.723	19.41	0.913
M2-L3	455,720	21,150	0.694	14.97	1.132
M2-L4	451,620	21,150	0.712	16.45	1.119
M3-1	445,260	30,500	0.748	30.88	0.565
M3-L2	445,260	30,500	0.687	16.55	0.744
M4-1	492,190	29,620	0.789	16.75	0.828
M4-L2	492,190	29,620	0.781	16.75	0.799
M5-1	463,830	39,780	0.980	37.33	0.521
M5-L2	463,830	39,780	0.866	37.50	0.619
M5-L3	463,830	39,780	0.975	45.50	0.524

Table 4 – Variability of semi-adiabatic calorimetry tests

Tests Compared			Percent Difference			Temperature Difference (°C)
			α_u	τ	β	
M1-1	versus	M1-2	9.3	3.9	4.3	6.2
M1-1	versus	M1-L3	6.7	33.0	9.8	3.2
M2-1	versus	M2-L3	8.8	17.5	19.0	3.6
M2-2	versus	M2-L4	1.5	15.2	22.5	5
M3-1	versus	M3-L2	8.2	46.4	31.7	3.5
M4-1	versus	M4-L2	1.0	0.0	3.5	1.9
M5-1	versus	M5-L2	11.6	0.4	18.8	8.1
M5-1	versus	M5-L3	0.4	21.9	0.4	1.8

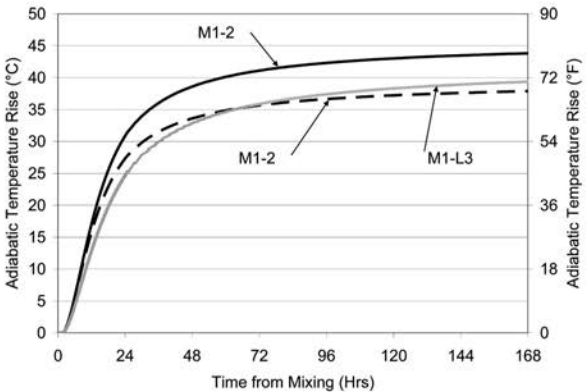


Figure 1—Adiabatic temperature rise for M1.

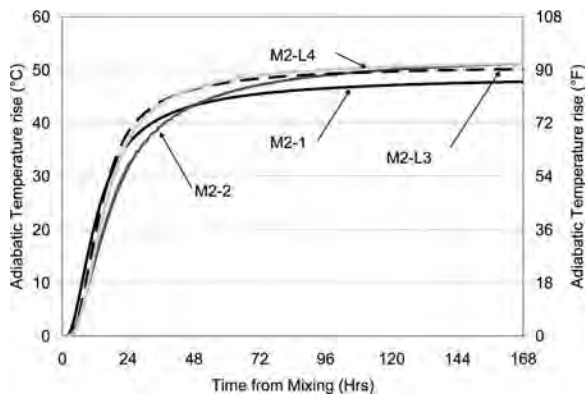


Figure 2—Adiabatic temperature rise for M2.

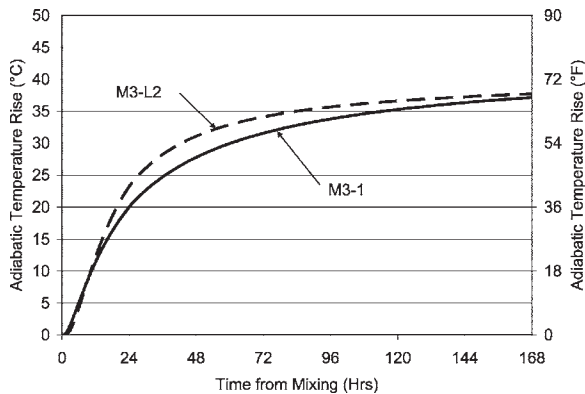


Figure 3—Adiabatic temperature rise for M3.

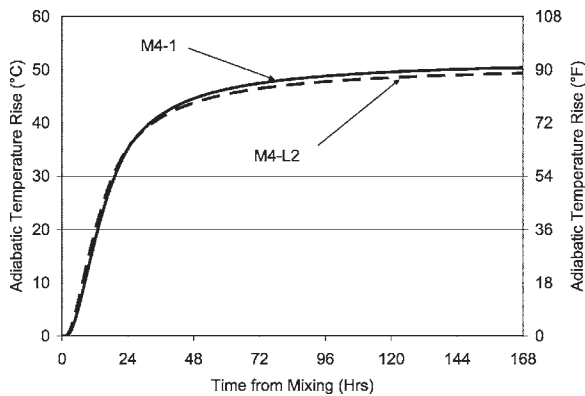


Figure 4—Adiabatic temperature rise for M4.

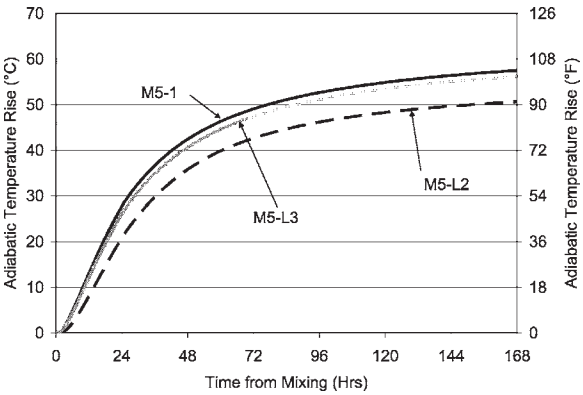


Figure 5—Adiabatic temperature rise for M5.

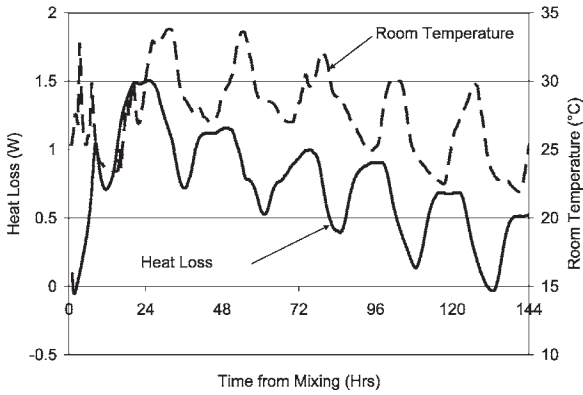


Figure 6—Heat flux and room temperature measured during test M3-1.

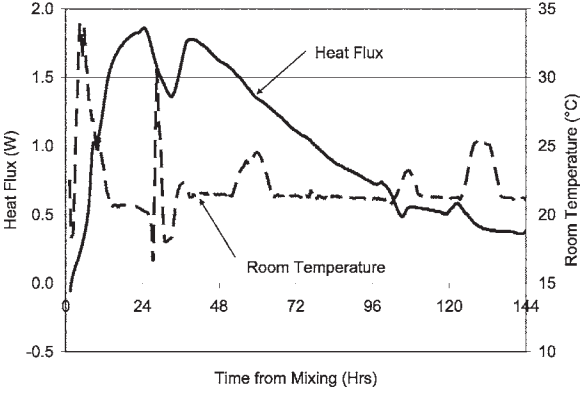


Figure 7—Heat flux and room temperature measured during test M5-1.

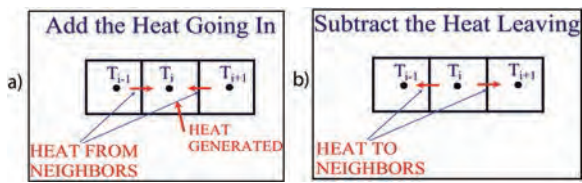


Figure 8—Control volume method for calculating concrete heat transfer.

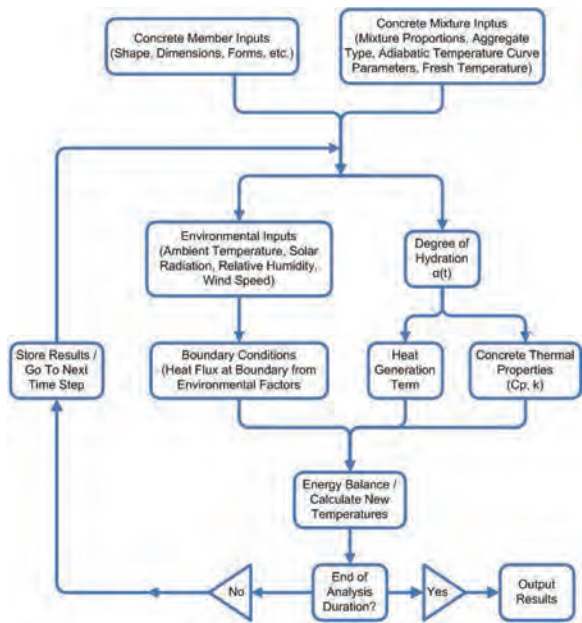


Figure 9—Flow chart of concrete temperature calculations.

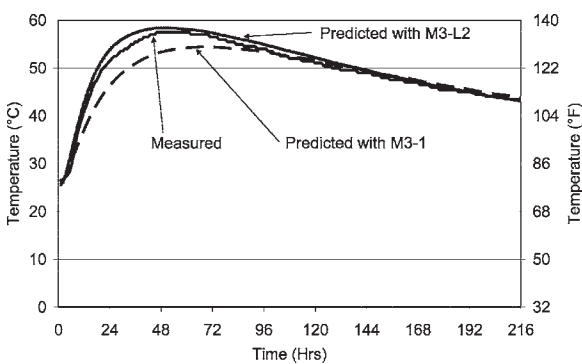


Figure 10—Concrete footing core measured versus predicted temperature using M3-1 and M3-L2.

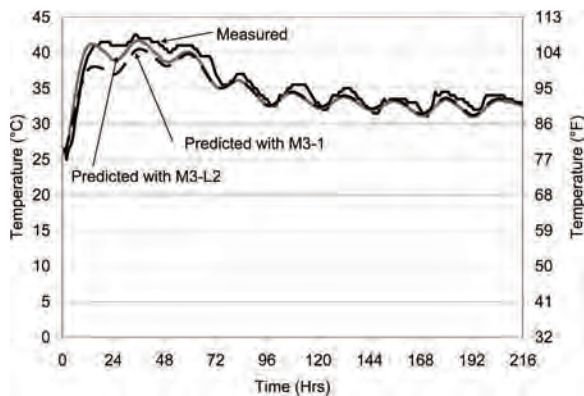


Figure 11—Concrete footing near surface measured versus predicted temperature using M3-1 and M3-L2.

Use of Thermal Measurements to Detect Potential Incompatibilities of Common Concrete Materials

by V.T. Cost and G. Knight

Synopsis: Abnormal early hydration resulting from “incompatibilities” of common concrete materials can result in erratic set and strength gain behavior and associated finishing, curing, and cracking issues. Contributing influences include high temperatures, cement sulfate levels, Class C fly ash content, chemical admixture use, and design approaches for retardation of hot-weather concrete.

Simple, expedient test methods are needed to identify potentially incompatible materials and conditions and to verify appropriate modifications to concrete proportions. Thermal measurements of the early heat development of materials mixtures in the laboratory (semi-adiabatic calorimetry) have been shown very useful toward this end.

Abnormal set and strength development of field concrete was reproduced in laboratory paste and mortar mixtures and studied using thermal measurements, verified by parallel mortar cube strengths. Sensitivities of various contributing influences were documented in extensive testing. Changing one or more of the key material or mixture characteristics was usually successful in restoring normal behavior. Recommendations are presented for avoiding related field issues and for the use of calorimetry testing programs for diagnosis of such problems.

Keywords: aluminates; calorimetry; cement-admixture interaction; exotherm; hydration heat; incompatibility; retardation; silicates; sulfates

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INTRODUCTION

Many concrete producers and contractors around the US, but especially in the deep south, have at some time experienced hot-weather concrete with unexpectedly extended set or perhaps even flash set before finishing could be done, followed by virtually no significant strength gain for a number of days. Such behavior has frequently gone undiagnosed, especially if the symptoms have disappeared after only a brief performance excursion.

In recent years these issues have been further complicated by more complex mix designs with more aggressive cement replacement and chemical admixture use. These occurrences have at times resulted in significant economic and scheduling disruptions, finishing and cracking concerns, torn out concrete, and litigation, often with little technical resolution. This study has been undertaken in an attempt to demonstrate simple and reproducible test methods for checking the interaction behavior of specific materials, to identify and quantify the most prevalent influences contributing to these issues, and to develop recommended practices for minimizing abnormal behavior in the field.

BACKGROUND AND LITERATURE REVIEW

Unexpected and undesired early interactions between cementitious ingredients and chemical admixtures have been documented and explored by various authors and researchers. The term “incompatibility” has been applied to a variety of different types of such abnormal performance in both plastic and hardened concrete, including setting and strength gain issues, problems with the air void system, and early age cracking. This paper will focus only on issues relating to set and strength gain. It is possible for a combination of common materials, each of which individually meets applicable specifications and has been used without problems in other mixtures or under slightly different conditions, to result in completely unexpected and abnormal behavior. The mixture in question may represent commonly used proportions, yet result in severely abnormal slump loss or setting tendencies (usually extended delays but occasionally flash set) followed by extremely poor early strength development. Late age strengths of such mixtures may be relatively unaffected though in more extreme cases even 28-day strengths can be significantly lower than expected. Such behavior has sometimes occurred in a concrete mixture that performed normally on a project for a period of time and then suddenly experienced these issues though no material source was changed. This

suggests that the threshold of incompatibility can be suddenly crossed due to slight, routine variability of one or more materials, methods, or other influences.

Some published articles pertaining to these issues are referenced. Some of the individual factors that influence incompatibility are well documented in these resources and some resolution approaches are offered. In order to determine the most practical and economical course of action for preventing or dealing with these abnormal behaviors, however, the concrete producer needs a better understanding of all of the significant (controllable) contributing factors and the relative importance of each.

Effects of cement sulfate level

Researchers who have investigated the possible causes of such concrete behavior have often cited under-sulfated cements as a major contributor, especially in the presence of some chemical admixtures. Cement manufacturers add soluble sulfate compounds to cement through the addition of gypsum and related materials in the finish grinding mills. Sulfates serve to facilitate normal set of cement by controlling aluminate hydration and they also enhance C_3S hydration. Uncontrolled initial aluminate reactions due to insufficient sulfate availability can result in rapid loss of concrete workability accompanied by excessive hydration heat and even flash set. Yet for reasons not completely understood, insufficient sulfates have also been found to influence extreme set delays^{1,2}. Such issues were reported as early as the 1960s^{3,4}.

Different forms of sulfates present in cement exhibit different rates of solubility. Of the most common forms, plaster (hemihydrate) is more readily soluble than gypsum (dihydrate). It is generally recognized that some balance of plaster and gypsum achieves the best performance, and this may be controlled to some extent through control of finish mill temperatures (hotter mill temperatures dehydrate more gypsum to plaster), though this opportunity may be limited. Increasing plaster content may help control initial C_3A hydration in complex mixtures but too much plaster may bring about false set (sometimes called plaster set) tendencies. Small increases of overall sulfate level have successfully addressed some incompatibility issues when insufficient soluble sulfates were determined to be the problem¹. The early reactions that take place in fresh concrete are quite complex and can be upset by the chemistry of admixtures and fly ash as well as higher temperatures. For optimum concrete performance, the proper balance of sulfate content and form would really need to be tailored for use in a particular project⁵. Of course, this is not usually practical and the cement manufacturer generally selects a single, constant sulfate level for “typical” applications.

There are many factors that influence what the ideal sulfate level of a cement should be. Some of the most critical are C_3A and alkali content, particle size distribution, and resulting sulfate forms. Some of today’s cements may be made with clinkers higher in alkali sulfates than in years past⁶, and lower portions of measured cement SO_3 may be readily soluble and available for control of initial aluminate reactions. For this and other reasons it is not uncommon for two similar cements that both perform acceptably to have somewhat different sulfate levels. Sulfate content may most often be optimized according to ASTM C 563, the basis of which is one-day strength of a standard mortar

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mixture. Since this process neglects the various materials and project influences that tend to increase sulfate demand in “real world” concrete, researchers have often questioned the wisdom of use of this test alone as the basis for selecting a sulfate content^{1,5}. While increasing sulfate level may improve the performance of concrete containing admixtures and fly ash, this must be done with caution since excessive sulfate can have damaging effects on concrete strength, shrinkage, and potentially harmful expansion. ASTM C 150 restricts sulfate content according to cement type, though sulfate levels higher than the “soft” limits may be used to the extent that measured mortar bar expansion in ASTM C 1038 testing does not exceed limits. To assure specification compliance, a practical sulfate level for the manufacturer to target would necessarily be somewhat lower than a value limited by expansion testing, recognizing normal production variability.

Fly ash issues

The use of Class C fly ash in concrete has been shown to be a potentially significant influence on erratic and abnormal interactions because C ash may contain relatively high levels of certain aluminate compounds, including C_3A . Since cement sulfates are theoretically optimized for control of the cement’s C_3A content, if fly ash that replaces some of the cement contains significantly higher levels of C_3A than the cement, it follows that the system may now be under-sulfated. Abnormal interactions that accompanied Class C fly ash replacement of cement at traditional levels were documented by several researchers^{1,5,7}. Since Class C ash replacement rates are often increased by the concrete producer in hot weather, taking advantage of this material’s tendency to retard set slightly, potential incompatibility may become even more likely. It may also seem counter-intuitive to the concrete producer that cement replacement with set-retarding fly ash could result in flash set. Fly ash use is an important economic and performance factor for many producers, so information on the relative sensitivity of fly ash content to incompatibility behavior is needed.

Temperature effects

Hot weather and higher fresh concrete temperatures are important influences on the potential for adverse reactions, with abnormal behavior becoming more likely with increases in material temperatures. Reported experiences with extreme incompatibility in the southern US have occurred almost exclusively in very hot weather, and some material combinations that have performed acceptably in moderate temperatures have been problematic in hotter weather periods. A major influence is that solubility rates of the most readily soluble sulfate forms decrease at higher temperatures⁸, though some other influences related to hot weather also tend to compound effects – greater use of fly ash, higher dosages of admixtures for retarding effects, and accelerated reactions.

Although some researchers have noted temperature effects on abnormal interactions or at least encouraged the use of representative concrete field temperatures in lab experiments, the relative influence of specific material temperatures or small changes in temperature may not have been widely explored or documented. This may in part be due to the fact that field concrete temperature control is expensive and, at a minimum, may be somewhat impractical as an incompatibility prevention measure. A better understanding of potential