Developing a Simple and Rapid Test for Monitoring the Heat Evolution of Concrete Mixtures for Both Laboratory and Field Applications

National Concrete Pavement Technology Center

Final Report
January 2006

Sponsored by
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16. Abstract

Various test methods exist for measuring heat of cement hydration; however, most current methods require expensive equipment, complex testing procedures, and/or extensive time, thus not being suitable for field application. The objectives of this research are to identify, develop, and evaluate a standard test procedure for characterization and quality control of pavement concrete mixtures using a calorimetry technique.

This research project has three phases. Phase I was designed to identify the user needs, including performance requirements and precision and bias limits, and to synthesize existing test methods for monitoring the heat of hydration, including device types, configurations, test procedures, measurements, advantages, disadvantages, applications, and accuracy. Phase II was designed to conduct experimental work to evaluate the calorimetry equipment recommended from the Phase I study and to develop a standard test procedure for using the equipment and interpreting the test results. Phase II also includes the development of models and computer programs for prediction of concrete pavement performance based on the characteristics of heat evolution curves. Phase III was designed to study for further development of a much simpler, inexpensive calorimeter for field concrete. In this report, the results from the Phase I study are presented, the plan for the Phase II study is described, and the recommendations for Phase III study are outlined.

Phase I has been completed through three major activities: (1) collecting input and advice from the members of the project Technical Working Group (TWG), (2) conducting a literature survey, and (3) performing trials at the CP Tech Center's research lab. The research results indicate that in addition to predicting maturity/strength, concrete heat evolution test results can also be used for (1) forecasting concrete setting time, (2) specifying curing period, (3) estimating risk of thermal cracking, (4) assessing pavement sawing/finishing time, (5) characterizing cement features, (6) identifying incompatibility of cementitious materials, (7) verifying concrete mix proportions, and (8) selecting materials and/or mix designs for given environmental conditions. Besides concrete materials and mix proportions, the configuration of the calorimeter device, sample size, mixing procedure, and testing environment (temperature) also have significant influences on features of concrete heat evolution process. The research team has found that although various calorimeter tests have been conducted for assorted purposes and the potential uses of calorimeter tests are clear, there is no consensus on how to utilize the heat evolution curves to characterize concrete materials and how to effectively relate the characteristics of heat evolution curves to concrete pavement performance. The goal of the Phase II study is to close these gaps.

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DEVELOPING A SIMPLE AND RAPID TEST FOR MONITORING THE HEAT EVOLUTION OF CONCRETE MIXTURES FOR BOTH LABORATORY AND FIELD APPLICATIONS

Final Report
January 2006

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Special thanks are extended to the technical working group (TWG) members: Gary Knight (Holcim), Paul Sandberg (WR Grace), Wes Woytowich (Lafarge), Peter Taylor (CTL), Anton Schindler (Auburn University), Todd Hanson (Iowa DOT), Gary Crawford (FHWA), and Leif Wathne (FHWA).Valuable input on the needs, importance, and current practices of various calorimeter tests provided by the TWG members are especially acknowledged.

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EXECUTIVE SUMMARY

The hydration of cementitious materials in a concrete mixture results in a number of exothermic chemical reactions which liberate heat. The heat generation process reflects the characteristics of concrete materials and mix proportions as well as the changes in construction and environmental conditions. This process also directly influences concrete workability, setting behavior, rate of strength gain, and pore structure development. Various test methods are available for measuring heat of cement hydration; however, most existing methods require expensive equipment, complex testing procedures, and/or extensive time, thus not being suitable for field application. The objectives of this research are to identify, develop, and evaluate a standard test procedure for characterization and quality control of pavement concrete mixtures using a calorimetry technique.

This research project contains three phases. Phase I was designed to identify the user needs, including performance requirements and precision and bias limits, and to synthesize existing test methods for monitoring the heat of hydration, including device types, configurations, test procedures, measurements, advantages, disadvantages, applications, and accuracy. Phase II was designed to conduct experimental work to evaluate the calorimetry equipment recommended from the Phase I study and to develop a standard test procedure for using the equipment and interpreting the test results. Phase II also includes the development of models and computer programs for prediction of concrete pavement performance based on the characteristics of heat evolution curves. Phase III was designed to study for further development of a much simpler, inexpensive calorimeter for field concrete. In this report, the results from the Phase I study are presented, the plan for the Phase II study is described, and the recommendations for Phase III study are outlined.

Phase I has been completed through three major activities: (1) collecting input and advice from the members of the project Technical Working Group (TWG), (2) conducting a literature survey, and (3) performing some trial tests at the CP Tech Center's research lab. The research results indicate that in addition to predicting maturity/strength, concrete heat evolution test results can also be used for (1) forecasting concrete setting time, (2) specifying curing period, (3) estimating risk of thermal cracking, (4) assessing pavement sawing/finishing time, (5) characterizing cement features, (6) identifying incompatibility of cementitious materials, (7) verifying concrete mix proportions, and (8) selecting materials and/or mix designs for given environmental conditions. Besides concrete materials and mix proportions, the configuration of the calorimeter device, sample size, mixing procedure, and testing environment (temperature) also have significant influences on features of concrete heat evolution process. The research team has found that although various calorimeter tests have been conducted for assorted purposes and the potential uses of calorimeter tests are clear, there is no consensus on how to utilize the heat evolution curves to characterize concrete materials and how to effectively relate the characteristics of heat evolution curves to concrete pavement performance. The research team believes that the goal of the Phase II study is to close these gaps.

The goal of the proposed Phase II is to conduct a more focused systematical study that brings test equipment and procedure development, heat evolution curve characterization, pavement performance prediction, and test/equipment specifications all together.

INTRODUCTION

Background and Problem Statement

Concrete quality control during paving is critical to ensure a desired pavement performance. Numerous test methods have been developed and are routinely used for control of field concrete quality including slump, air content, and strength. However, there are currently no standard methods that can both practically and accurately monitor the hydration process of field concrete–a property known to be very influential on not only early-age behavior, but long-term performance. Lately, maturity testing has been increasingly used by state Departments of Transportation (DOTs) to monitor strength development of field concrete with time. Although concrete maturity is related to the temperature development in concrete with time, this measurement, or the maturity value, does not describe the cement hydration process.

The hydration process is important because it reflects the characteristics of concrete materials and mix proportions as well as the changes in construction and environmental conditions. The hydration process also directly influences concrete workability, setting behavior, strength gain rates, and pore structure development. Therefore, proper information on the hydration process of field concrete may be used to control field concrete quality and/or to predict the concrete performance.

The hydration of cementitious materials in a concrete mixture results in a number of exothermic chemical reactions which liberate heat. The hydration process can be monitored by measuring the total liberated heat (via temperature changes) over time. Research has demonstrated that the rate and amount of heat liberated from hydration greatly depends on the chemical compositions and physical properties of the cement, supplementary cementitious materials (SCM), and chemical admixtures. Both the concrete mix proportions and curing temperature play important roles as well. As a result of these sensitivities, deviations in the quantities or characteristics of the concrete constituents, especially the type and amount of cementitious materials used, can be readily detected by monitoring the heat of hydration (PCA 1997).

The heat of hydration can be determined by various means. Traditionally, it has been determined by measuring the heat of the solution (*ASTM C186*). More recently, the calorimetry test is increasingly used because it monitors heat of hydration with time. This procedure has also been termed a heat signature or thermal fingerprint test since it measures a temperature-related property that is unique to a given concrete mix. From a purely theoretical standpoint, there are two major types of calorimetry tests: isothermal and adiabatic. Using isothermal calorimetry, the heat of hydration is measured by monitoring the heat flow from the specimen while both the specimen and the surrounding environment are maintained at approximately the same temperature. In an adiabatic calorimetry test, the heat of hydration is measured by monitoring the heat flow from the specimen while the specimen is under an insulated condition and has no or very little heat loss. Since a “perfect” isothermal or adiabatic calorimetry test is nearly impossible to conduct without delicate (and thus expensive) equipment, a hybrid termed semi-adiabatic calorimetry testing is more frequently employed.

To date, a great deal of work has been done using calorimeter techniques in research labs. The test results reveal not only the effects of cementitious materials and/or admixtures on hydration but also the cement-admixture compatibility (Nocuñ-Wczelik 2001, Lawrence et al. 2003, Sandberg 2004). However, while there has been a good deal of testing performed to date, there continues to be no standardized test method for controlling and monitoring the heat of hydration of field concrete.

Part of the reason is that most of the currently available equipment for calorimetry testing is expensive and complex, and thus not suitable for field application. In addition, a conventional adiabatic test generally takes as long as a week to complete. A shorter test period is needed to make this test applicable to field monitoring of concrete mixtures. Furthermore, many practical engineers report finding it difficult to interpret the meaning of the heat evolution curve correctly and effectively. Therefore, while there are a number of devices that measure the hydration process, there remains a need for identification of a relative rapid and inexpensive test, establishing a specification suitable for AASHTO, ASTM, and/or state DOT procedures. The need for standardization of concrete calorimeters and its urgency has been addressed in *Concrete Pavement Technology Long Term Research and Technology Plan (Task 15)* (Harrington). It is believed that if a rapid and inexpensive test for monitoring and controlling hydration is developed, the quality control of field concrete will be substantially improved.

Research Objectives

The aim of the present research project is to identify and develop a standard test procedure for monitoring paving concrete using a calorimetry technique. This research has three specific objectives:

- To identify and synthesize existing test procedures for measuring the heat of hydration of concrete using calorimetry tests and related methods
- To identify, refine, and/or develop a relatively simple, inexpensive device and a prototype test procedure for measuring the heat of hydration of field concrete
- To develop a model and software for converting the raw test data into relevant test metrics as well as acceptance criteria for test results

It is expected that the research result will be a new and important tool for concrete mix characterization and quality control.

A collaborative research team has been formed, consisting of members from Iowa State University (ISU) and The Transtec Group. A technical working group (TWG), consisting of experts from academia, the Federal Highway Administration (FHWA), state DOTs, and industry, has been created to oversee the work conducted under this project.

Research Approach and Scope

This research project consists of three phases:

- Phase I was to conduct a literature and experts survey, identifying the user needs for a calorimeter test and synthesizing existing test methods for measuring the heat of hydration. It started in October 2004 and was completed in August 2005.
- Phase II is to bring test equipment and procedure development, heat evolution curve characterization, pavement performance prediction, and test/equipment specifications all together.
- Phase III study for further development of a much simpler, inexpensive calorimeter for field concrete was also proposed in order to further implement the Phase II research results.

Summary of Phase I Study

The project started with a kickoff meeting on October 1, 2004. All TWG and research team members attended the meeting. The TWG members included Gary Knight (Holcim), Paul Sandberg (WR Grace), Wes Woytowich (Lafarge), Peter Taylor (CTL), Anton Schindler (Auburn University), Todd Hanson (Iowa DOT), and Gary Crawford and Leif Wathne (FHWA). Valuable input on the needs, importance, and current practices of various calorimeter tests were provided by the TWG members at the meeting. Broad discussions were held that addressed specific issues on the product development (i.e., product configuration, cost, testing procedure, result interpretation and application) This input and discussion has been summarized and considered carefully by the research team members in their recommendations for the new device development of the phase II study.

In Phase I, a collaborative research team consisting of members from the CP Tech Center, Iowa State University (ISU), and the Transtec Group, completed the following two major tasks:

1. Identified the user needs for a calorimeter test, including performance requirements, and precision and bias limits
2. Identified and synthesized existing test procedures for measuring the heat of hydration of concrete using calorimetry and other methods, including efforts both in the U.S. and abroad

These tasks have been accomplished through three major activities: (1) collecting input and advice from the project Technical Working Group (TWG), (2) conducting a literature survey, and (3) performing some trial tests at the CP Tech Center's research lab.

A literature review on heat evolution tests was conducted and the results were synthesized to provide the following information:

- Factors affecting the concrete heat evolution
- Existing devices and test methods for heat evolution measurement (type of calorimeter, configurations, procedures, measurements, advantages, disadvantages, applications, and accuracy)
- Existing temperature sensors/datalogger (temperature range and sensitivity)
- Existing models for predicting heat of hydration and interpreting raw data
- Potential applications of the test results (such as predicting concrete set time, sawing/finishing time, identifying incompatibility problems, and checking cement characteristics – various sulfate phases)

A series of mini-tests were performed at the CP Tech Center using the different Dewar devices to investigate effects of device insulation, sample size, curing temperature, and mineral admixtures on heat evolution curves.

In the following sections, the major findings from the Phase I study are briefly summarized, the modified plan for Phase II is described, and a potential Phase III study is recommended.

LITERATURE REVIEW

This section includes the literature review of the hydration of the cementitious materials, existing devices and test methods for heat evolution measurement, existing temperature datalogger and sensors, existing models for predicting heat of hydration, and potential uses of heat signature testing.

Cement Hydration

Cement hydration is a complex process and can be influenced by several factors. This section discusses the heat of hydration and factors that influence the rate and amount of heat liberated from hydration of the cementitious materials.

Heat of Hydration

As shown in Figure 1, cement hydration process is typically divided into five stages. As soon as cement is mixed with water, a period of rapid heat evolution (stage 1) occurs and lasts about fifteen to thirty minutes. The rapid reactions result from ions dissolving in water and reacting between C3A and gypsum. This stage is normally not captured by the calorimeter test due to its short reaction time. The heat evolution curves generally measured begin with the dormant period of cement hydration (stage 2). During the dormant period, cement hydration ceases, little heat is generated, and the concrete is flowable. As ion dissolution continues with time, the ion concentrations of C3S and C2S increase in the concrete system. This period generally lasts less than five hours. At the end of the dormant period, the significant hydration starts again due to C3S and C2S hydration (stage 3). This period of cement hydration is called the acceleration period. Concrete temperature increases rapidly during this period. As time increases, the rate of heat generation gradually slows (stage 4). In this phase, the thickness of the hydrate layer that covers unhydrated particles increases, and the surface area of the unhydrated parts decreases. The layer of cement hydrates acts as the diffusion area, which governs the permeability of the water and dissolved ions. Finally, cement hydration reaches the steady state (stage 5). Both stages 4 and 5 are known as the diffusion control phase.

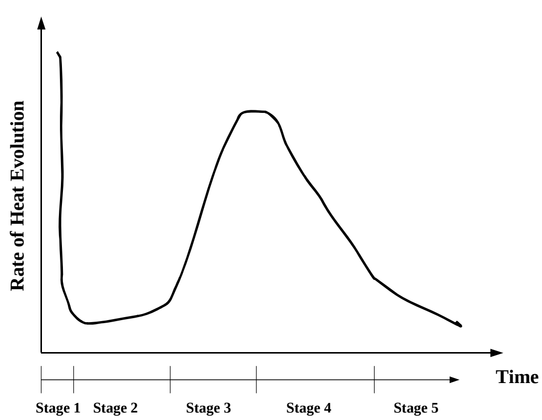

Figure 1. Cement hydration process

Factors that Affect the Heat of Hydration

The rate and amount of the heat liberated greatly depends on the chemical and physical properties of the cement, water/cement ratio, supplementary cementitious materials (SCM), chemical admixtures, and curing conditions. These factors are discussed in the following section.

<u>Cement Type</u>

The heat generation rates for different types of cement under the same condition, which can be measured as a rise in temperature, vary significantly. In Figure 2, the temperature rise for Type III cement is about 45°C. However, the increase for Type IV cement is only 25°C. The difference in heat generation rates for various cements is mainly due to the chemical composition and the cement fineness.

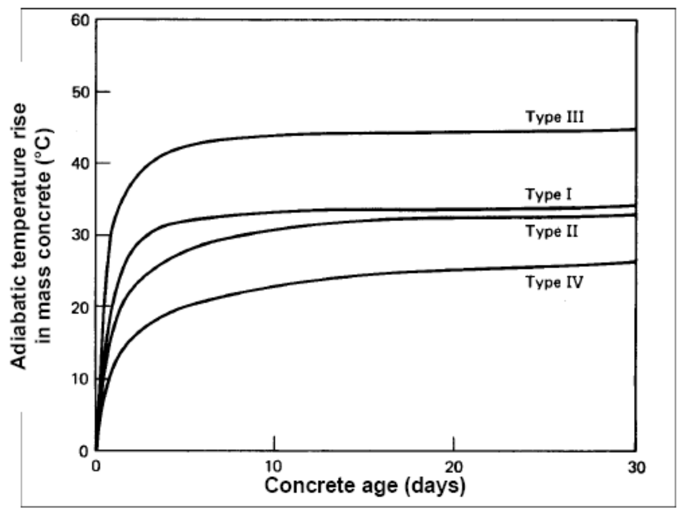

Figure 2. Temperature increase of mass concrete under adiabatic conditions (adapted from Mindess and Young 1981)

The effect of chemical composition can be identified by evaluating the rate of hydration of the individual compound and its percentage in the cement. Each chemical compound shows a different hydration rate and total liberated heat. The hydration characteristics of cement compounds are listed in Table 1.

Table 1. Hydration characteristics of cement compounds

Compounds	Reaction rate	Amount of heat liberated	Contribution to cement heat liberation
C3S	Moderate	Moderate	High
C2S	Slow	Low	Low
$C_3A + C\bar{S}H_2$	Fast	Very High	Very High
$C_4AF + C\bar{S}H_2$	Moderate	Moderate	Moderate

The rates of the pure cement compounds are given in Figure 3a. It can be seen that C3A reacts the fastest, followed by C3S and C2S. The presence of gypsum slows the early age reaction of C3A. The hydration rate of the compounds in typical cement is plotted in Figure 3b. The figures show that C3S and C2S react more rapidly than they do in their pure pastes. C4AF falls between C3S and C2S. Both figures indicate that C3A and C3S are the most reactive compounds; their total liberated heat is also high.

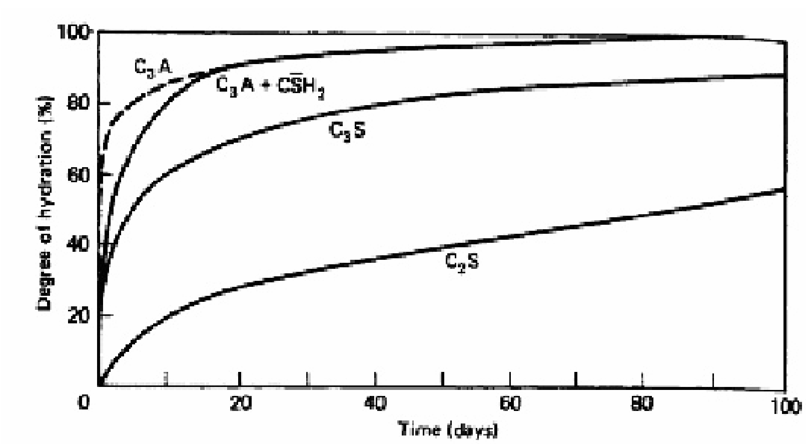

Figure 3a. Hydration rate of cement compounds—in pastes of pure compounds (adapted from Mindness and Young 1981)

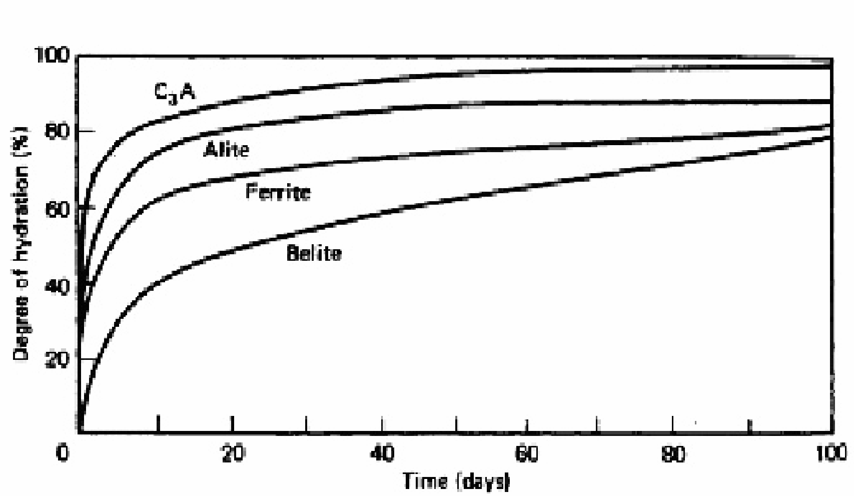

Figure 3b. Hydration rate of cement compounds—in a type I cement paste (adapted from Mindess and Young 1981)

Lerch and Bough (1934) studied the effect of C3A and C3S on the heat of hydration of pastes. Figure 4 indicates that C3A content significantly increased the rate and amount of generated heat. The total heat is almost doubled when the C3A increases from 0 to 20%. Regardless of the C3A content, the reaction is stable after sixteen hours. Figure 5 shows the effect of C3S, which is similar to C3A.

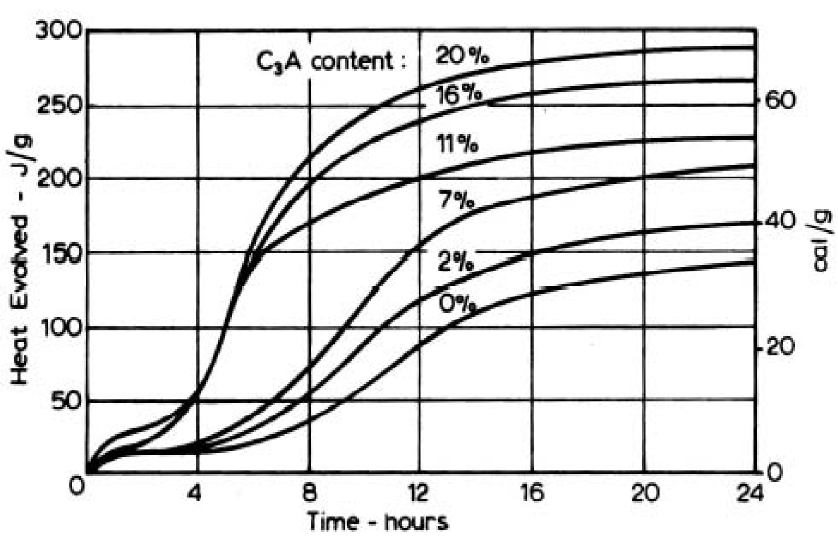

Figure 4. Effect of C_3A content ($C_3S \approx$ constant) on heat of hydration (adapted from Lerch and Bogue 1934)

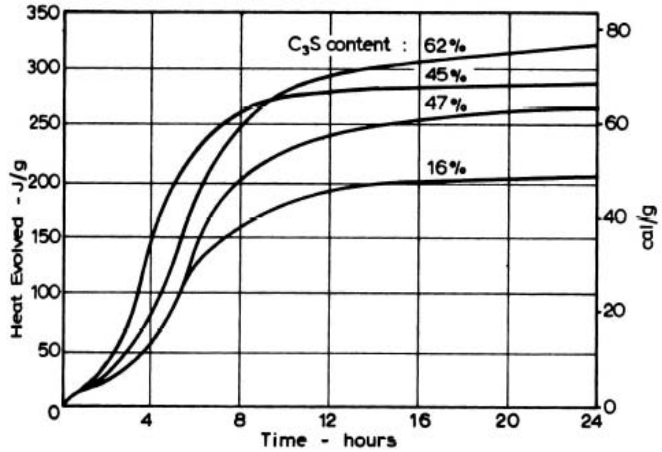

Figure 5. Effect of C_3S content ($C_3A \approx$ constant) on heat of hydration (adapted from Lerch and Bogue 1934)

Sulfate Content

During the final process of cement production, a small amount of gypsum is added and interground with the clinker to control the early reaction of tricalcium aluminate. With a low or over dosage of sulfate, the cement will have false or flash set. The proper amount of sulfate required for cement varies with cement composition and fineness.

Lerch (1946) conducted a series of tests to study the effect of gypsum on hydration in terms of the heat liberation. He found that heat liberated in 30 minutes, was reduced by increasing SO3 content regardless of the content of C3A (Figure 6). This finding could be explained by the theory that alumina is less soluble in a lime-gypsum solution than in limewater (Lerch et al. 1929). Adding gypsum saturates the solution more quickly and the reaction of C3A is retarded. Therefore, the heat of hydration is reduced.

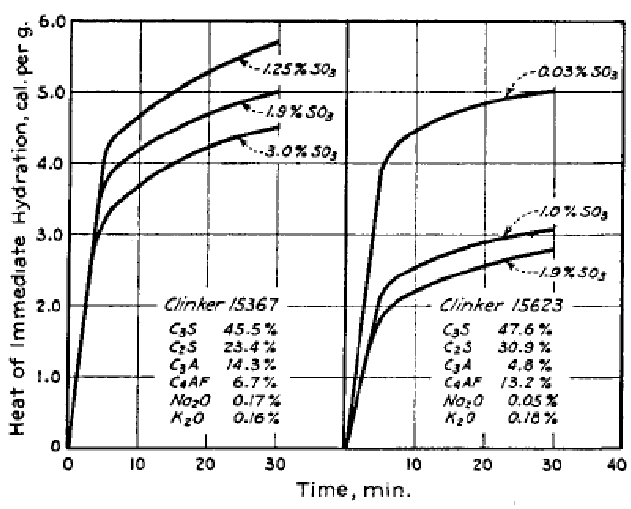

Figure 6. Heat of immediate hydration with SO_3 varied (adapted from Lerch et al. 1946)

Fineness

Fineness is another factor that can affect the hydration of cement. Fineness of cement affects the placeability, workability, and water content of a concrete mixture. It is normally measured in terms of specific surface area. The average Blaine fineness of modern cement ranges from 3,000 to 5,000 cm2/g. Although cement with different particle distribution might have the same specific surface area, the specific surface area is still considered to be the most useful measure of cement fineness.

Since hydration occurs at the surface of cement particles, finely ground cement will have a higher rate of hydration. It has a higher specific surface area, which means there is more area in contact with water. The finer particles will also be more fully hydrated then coarser particles. However, the total heat of hydration at very late ages is not significantly affected. Figure 7 shows how the fineness increases the early age heat of hydration for two different types of clinkers.

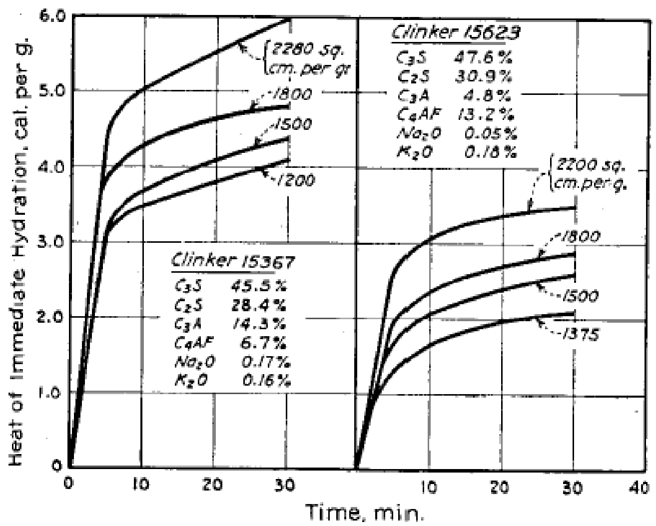

Figure 7. Heat of hydration with specific surface varied (adapted from Lerch et al. 1946)

Water/Cement Ratio

An important aspect during hydration is the decrease in porosity. The precipitated hydration products, which have lower specific gravities and larger specific volumes, cause cement grains to expand continuously as cement hydration proceeds. However, the volume of the hydration product is less than the total volume of the cement and water that reacted to form it. The hydration product will not fill the volume made available for it. If external water is available, the cement will hydrate continuously until either the cement is completely hydrated or until the available space within the paste is completely filled. Complete hydration of cement is generally assumed to require a water/cement ratio of about 0.4 (Van Breugel 1997). According to Young et al., hydration will stop when the amount of water is not enough to form a saturated C-S-H gel. A minimum w/c ratio of 0.42 is required for complete hydration. If external water is not available, the cement paste will dry as hydration proceeds. Additionally, when the internal relative humidity drops below about 80%, hydration will stop.

Cement with a high w/c ratio has more water and microstructural space available for hydration of cement, which in turn results in a higher ultimate degree of hydration. Since the heat of hydration is directly related to the degree of hydration, the heat generation is affected by the w/c ratio (Figure 8). The rate of heat evolution at early ages is not significantly affected by the w/c ratio. However, consistent with the findings of Byfors (1980), the heat evolution rate starts to decrease as the w/c ratio decreases after a certain time. In Figure 8, the total heat liberated increase more than 100 KJ/Kg when the w/c changes from 0.3 to 0.7.

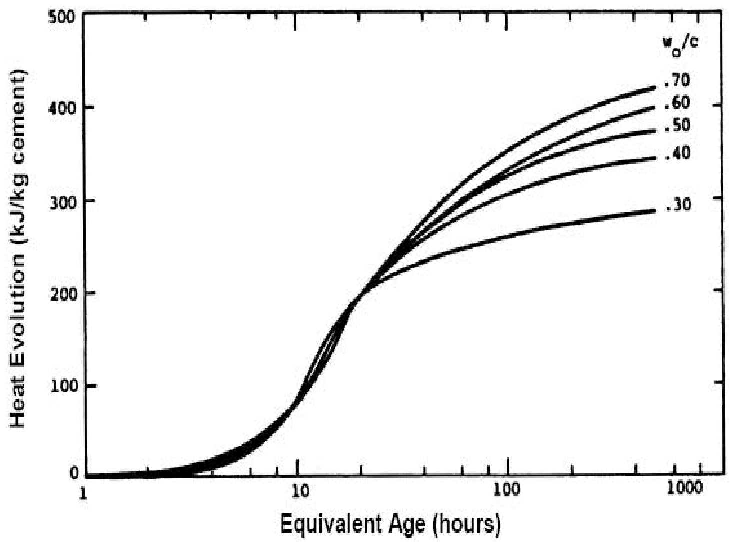

Figure 8. Effect of w/c ratio on the heat evolution (RILEM 42-CEA 1981)

Curing and Initial Temperature

Normally, concrete pavement is cast from spring to fall. During this period, the environmental temperature is completely different. Therefore, the environmental temperature needs to be considered. A number of investigators have studied the effect of curing temperature on cement hydration (Figure 9). Cement hydration is accelerated at early ages under high environmental temperature but decelerated later on. The initial hydration under high temperatures forms "shell," a coat layer of hydration products on the surface of the cement particles, which delays the continuation hydration. The shell is denser with increasing temperature. Cement hydration under lower temperatures is generally higher than cement hydration under higher temperatures. Blended cement with fly ash is similar to OPC. The initial reaction temperature has a similar effect on the rate of cement hydration. The higher the initial reaction temperature, the higher the hydration rate at early age. However, at later age, the hydration under lower initial reaction temperatures could be higher.

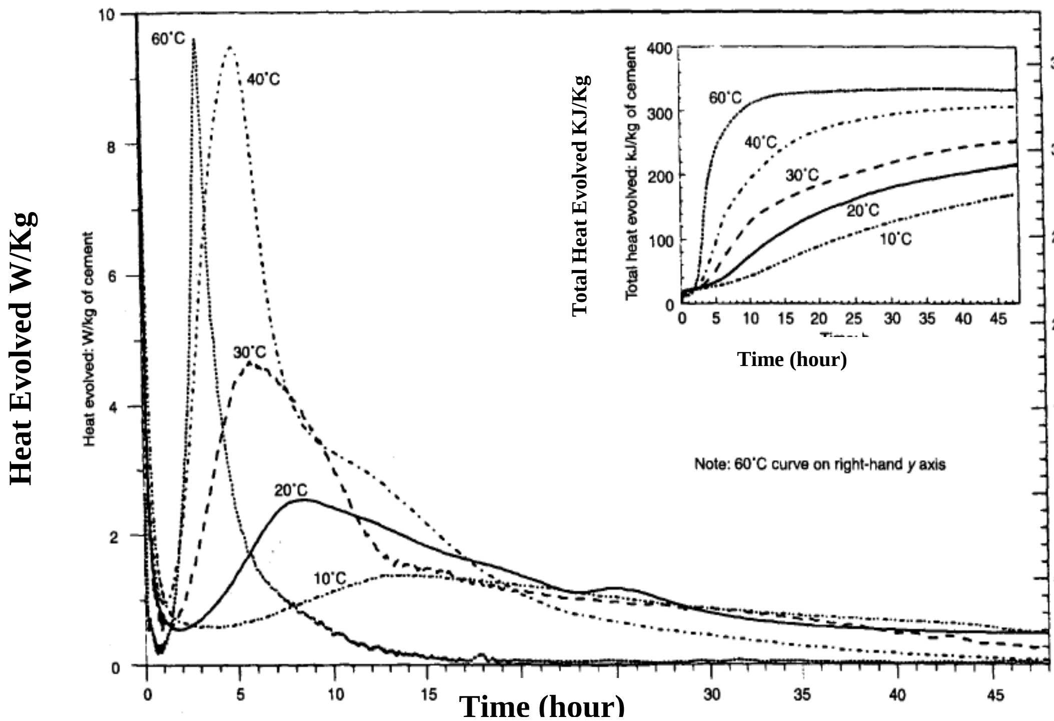

Figure 9. Effect of curing temperature on hydration
(adapted from Escalante-Garcia et al. 2000)

<u>Supplemental Cementitious Materials</u>

Supplemental cementitious material concrete normally displays slow hydration, accompanied by low temperature, slow setting, and low early age strength. This effect is more pronounced as the proportion of SCMs in the blended cement increases and when the concrete is cured at a low temperature. The properties of the SCM concrete are caused by the reduction of the cement content and also by the Pozzolanic reaction. It is generally accepted that the silicate and aluminate phase of SCMs react with Ca(OH)2 produced by cement hydration to form calcium silicate and aluminate hydrates (Lea 1970). This reaction is known as the Pozzolanic reaction (Equation 1).

$$Al/S + CH + H_2O \rightarrow CSH, \text{Al-phase} , \qquad (1)$$

The Pozzolanic reaction is slower than C3S hydration; however, the reaction rate is similar to C2S. As a result, the Pozzolanic reaction produces less heat than the cement hydration.

Fly ash

Various researchers have studied the effect of fly ash on cement hydration. Crow et al. (1981) determined that adding a low-calcium fly ash reduces the heat of hydration of cement Some high-calcium Class C fly ash with self-cementitious properties may react very quickly with water, releasing excessive heat just like normal OPC hydration (Joshi et al. 1997). The total heat of hydration of fly ash normally depends on the content of CaO. Schindler (2003) recommended the total heat be equal to 1,800 times the percent of CaO. Figure 10a shows that the addition of fly ash not only decreases the maximum heat generation rate but also postpones the peak of hydration. As the fly ash ratio increases, the peak becomes wider. Figure 10b shows that the retardation of cement hydration mainly occurs during the dormant and acceleration periods. When cement-fly ash cement mixes with water, the Ca2+ ion in pure solution is removed by the fly ash, which reacts like a Ca sink. The depressed Ca2+ concentration delays the nucleation and crystallization of CH and C-S-H, retarding hydration (Langan et al. 2002).

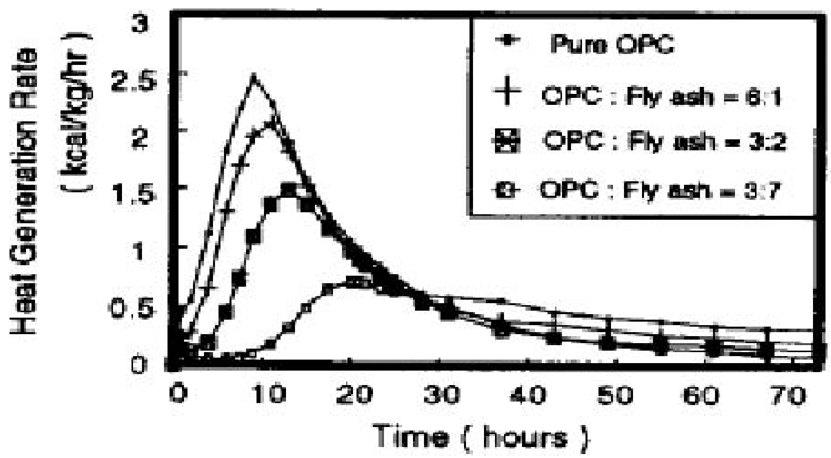

Figure 10a. Effect of class F fly ash on heat generation (adapted from Kishi and Maekawa 1995)

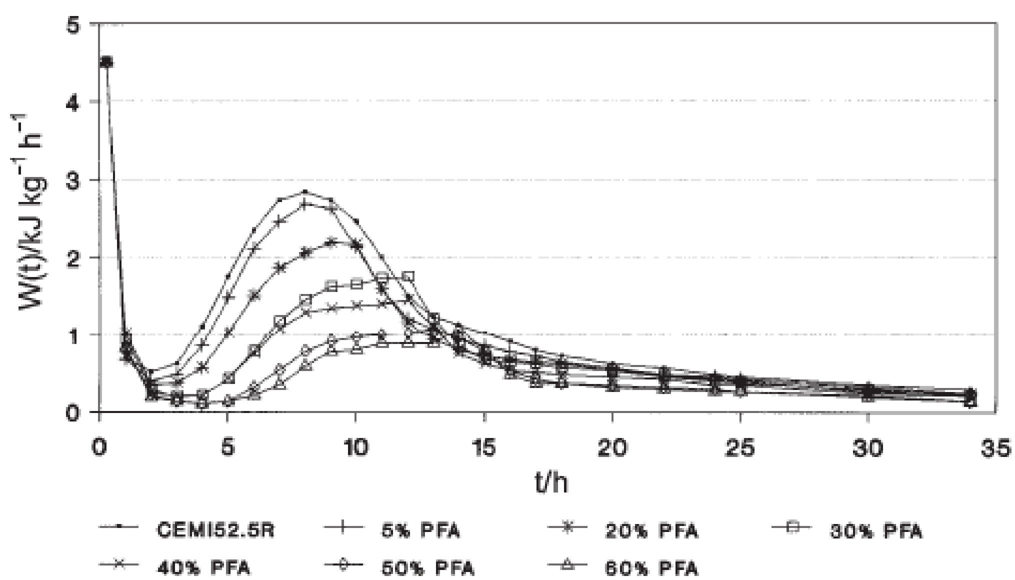

Figure 10b. Effect of class C fly ash on heat generation (adapted from Nocuñ-Wczelik 2001)

GGBF slag

Figure 11 shows the effect of different levels of slag on the heat generation ratio. There are two peaks for slag-blended cement; the first is caused by cement hydration and the second is due to slag reaction. The slag-blended cement reaches the first peak at the same time as the pure OPC, indicating that adding GGBF slag into concrete will not delay the reaction of cement. The second peak is unaffected by the amount of replacement slag. Kishi et al. (1994) explained that the slag can react independently as long as sufficient Ca(OH) is released from the cement hydration.

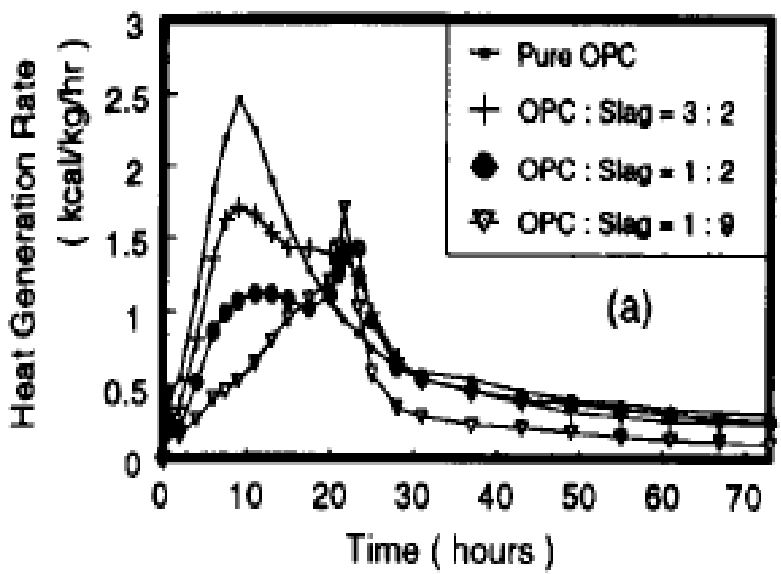

Figure 11. Effect of slag on hydration (adapted from Kishi and Maekawa 1994)

However, Hogan and Meusel (1981) found that the setting time of slag-blended cement is delayed; there is a ten to twenty minute delay for each 10% addition of slag. On the other hand, Uchikawa (1986) found that the peak of cement hydration was accelerated due to the finely ground slag's consumption of Ca2+ in the liquid phase when the fineness of the slag was increased (Figure 12).

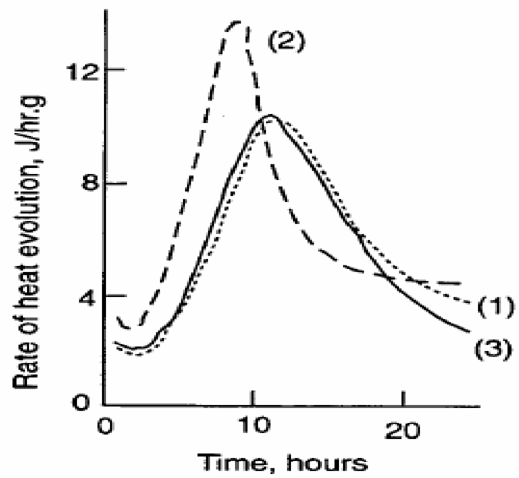

Figure 12. Heat evaluation at 20°C
(1) 40% coarse slag (400 m2/Kg) (2) 40% fine slag (592 m2/Kg) (3) OPC
(adapted from Uchikawa 1986)

Ma et al. (1994) studied the hydration of blended cement containing 65% slag at different temperatures ranging from 10°C to 55°C. In this study, the total heat of blended cement during the first 20 hours was significantly increased. The test results also showed that temperatures less than 40°C have little effect on the total heat. However, the total heat increases rapidly with temperatures above 40°C (Figure 13). Consistent with the findings of Klierger et al. (1994), these results indicate that the slag-blended cement has low reactivity at room temperature and is strongly heat activated.

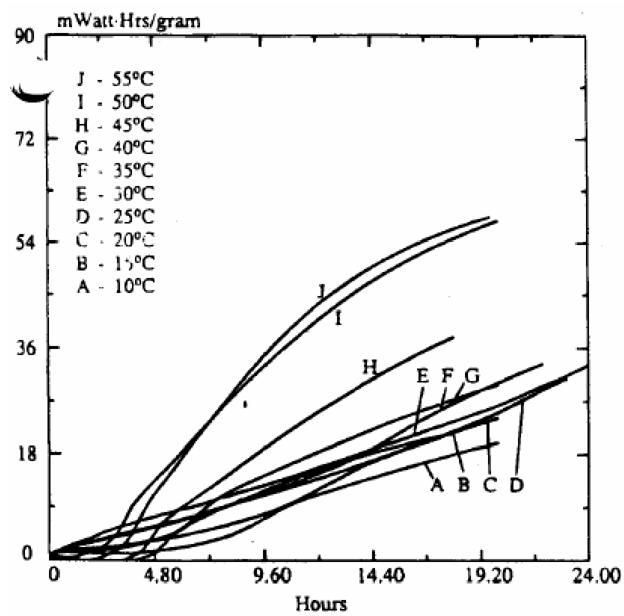

Figure 13. Total heat of blended cement with slag at different temperatures
(adapted from Ma et al. 1994)

Chemical Admixture

Chemical admixture is added to modify the setting time and reduce the water requirement of the concrete mix. Chemical admixtures may significantly change the cement hydration rate, and therefore, the heat generation. ASTM C 494 classified the chemical admixtures into the following seven types:

Type A—Water-reducing
Type B—Retarding
Type C—Accelerating
Type D—Water-reducing and retarding
Type E—Water-reducing and accelerating
Type F—Water-reducing high range
Type G—Water-reducing, high range, and retarding

Water reducers are defined as admixtures that decrease the requirement of water to achieve a given workability. They sometimes accelerate or retard the concrete setting. When a water reducer is used without changing the mix proportion, the heat of hydration and temperature rise of concrete at early age may increase. Using a water reducer can decrease the cement content at a given strength and slump, which in turn results in the reduction of heat generation and temperature rise (Massazza et al. 1980).

Retarder is an admixture that delays the setting and initial hardening of concrete. The ingredients of the retarder are similar to that of the water reducers. Ben-Bassat (1995) states that "the heat of hydration and temperature rise of concrete containing retarder are less at early age, and they are equal at about 3-7 days."

Accelerator is used to speed up the strength gain at early ages and reduce the setting time. Accelerating admixtures are normally used in a cold environment or where the early gain of strength and short setting time are required. An accelerator can increase the rate of heat evolution at the setting stage. Depending on the chemical composition of the accelerators, the rate of heat liberation in the hardening stage may also increase (Nagataki 1995).

High-range water-reducing agents (superplasticizers) are a new class of water-reducing agents. The workability of high-range, water-reducing agents increase more often than normal water-reducing agents. The use of superplasticizers mildly retards the setting of concrete, which in turn can reduce the heat generation in the setting period. However, superplasticizers do not affect the total heat of concrete. Figure 14 shows that the adding of superplasticizers reduces the adiabatic temperature at early age.

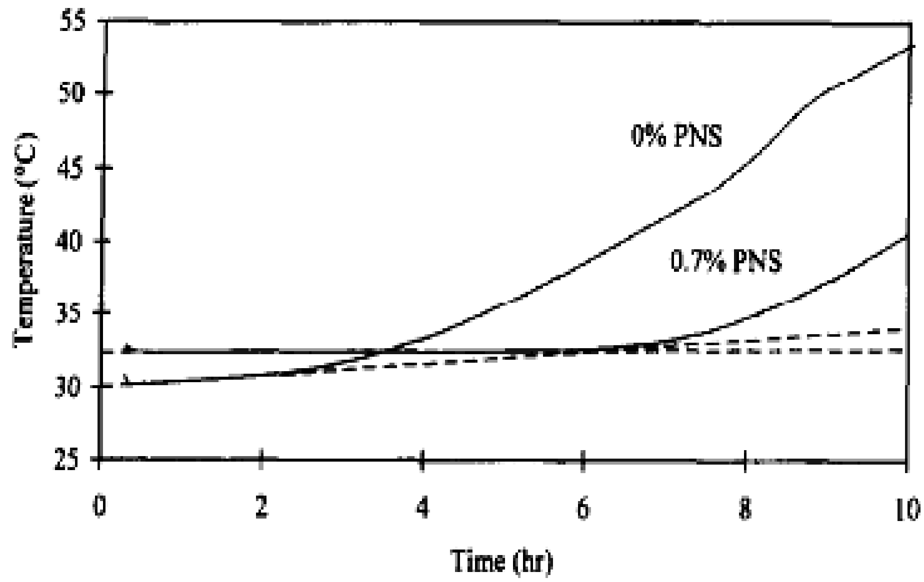

Figure 14. Effect of superplasticizer on cement hydration (adapted from Jolicoeur et al. 1998)

Existing Test Methods

Cement hydration is an exothermal process. The measurement of the heat is a convenient way to monitor and quantify the hydration reaction. There four major types of calorimeters used in cement science and technology: adiabatic calorimeters, semi-adiabatic calorimeters, isothermal calorimeters, and solution calorimeters. Expect for these four major types of calorimeters, other methods are developed to monitor the heat generation process.

Adiabatic and Semi-adiabatic Calorimeters

Since it is impossible to achieve an adiabatic environment, the calorimeter is considered to be adiabatic as long as the temperature loss of the sample is not greater than 0.02 K/h. The heat loss is prevented by controlling the temperature of the surrounding environment. The insulating materials could be water, air, and heated containers. Water insulation is the most popular way.

Adiabatic heat measurements are most convenient for producing a continuous heat of hydration curve under curing conditions close to mass curing. And also adiabatic hydration curves are the most suitable starting points for temperature calculation in hardening structures. One major drawback of an adiabatic calorimeter is that the effect of the curing temperature on the rate of hydration is measured implicitly. The activation energy is required to convert the results to the isothermal reference temperature. The results can also be affected by the assumption of the thermal properties of the materials. The advantage is that the heat evolution of an actual concrete mixture can be determined. Ballim (2004) conducted the adiabatic calorimeter test to obtain the rate of heat evolution and used this as an input to calculate the mass concrete temperature. De

Schutter (1995) and Tanaka et al (1995) performed the adiabatic tests to estimate heat of hydration of blast furnace slag blended cement.

Semi-adiabatic calorimeter is similar with the adiabatic calorimeter. But it allows a certain amount of heat loss to the environment. The maximum heat loss should be less than 100 J/(h·K). The calculated adiabatic curve from the semi-adiabatic test is lower than the curve from the true adiabatic test. Since the temperature development is lower than the sample in adiabatic test due to heat loss. Semi-adiabatic method is suitable for pasts, mortars, and concrete samples. It is been widely used for determining the heat signature of concrete.

The "Round-Robin" test program had been conducted by RILEM to compare the performance of different types of calorimeters. Fourteen different organizations participated in the program to compare the adiabatic curves and the predicted adiabatic temperature curves from semi-adiabatic calorimeter and to find the main factors that affect the results form different calorimeters. All tests used the same materials and mixing proportion. It is found that, for the adiabatic test, 50% of the adiabatic temperature rise variations are in a narrow range of only 2K and that the specimen size and the temperature do not significantly affect the temperature rise. For the semi-adiabatic test, the mean temperature rises are 2%–3% below the results from the adiabatic tests. This indicates that the semi-adiabatic calorimeter could be used to predict the adiabatic temperature rise. Table 2 shows the summary of the tests data.

Table 2. Variability of adiabatic and semi-adiabatic test results (adapted from P. Morabito 1998)

	Rise after 24 hours		**Rise after 48 hours**		**Rise after 72 hours**	
	Adiabatic	**Semi-adiabatic**	**Adiabati c**	**Semi-adiabati**	**Adiabatic**	**Semi-adiabatic**
Mean values (K)	36.7	35.9	43	41.6	44.7	44.6
Highest variability	+8.9%	+4.8%	+6.9%	+5.8%	+6.3	+4.4%*
Lowest variability	-13.2%	-12.5%	-10.3%	-12%	-9.6%	-4.8%*

* Calculated on four tests

Isothermal Calorimeter

Isothermal calorimeters are usually used for paste samples. These tests are conducted at a constant temperature. The heat of cement hydration is directly measured by monitoring the heat flow from the specimen. The total heat evolution can be determined by summating the measured heat over time. The disadvantage is that the duration of this test is normally limited to seven days due to the signal sensitivity limitations. Beyond this point, it is hard to distinguish the signal from its background. Also, the isothermal tests do not take into account the cement reactivity change due to the change of temperature. It is hard to predict the temperature increase of concrete from these results, since the conditions in the real structure where the temperature continually changes are not reflected.

Isothermal calorimeters are more widely used for studying the reaction of kinetics of pure cement pastes. Many researchers have applied this method to study the cement heat signature. Ma et al. (1994) conducted the isothermal calorimeter tests to study the hydration behavior of blended cements containing fly ash, silica fume, and granulated blast furnace slag over the temperature range of 10°C to 55°C. The relationship between the blended cement reactivates and the curing temperature was established. Xiong and Breugel (2001) used the 3114/3236 TAM Air isothermal calorimeter to determine the kinetics of cement hydration processes at different temperatures and applied the results to the numerical simulation model. Some other researchers also used this method to determine the activation energy of the cement.

Solution Calorimeter

ASTM C 186, Standard test method for heat of hydration of hydraulic cement, describes the test of measuring cement heat of hydration using a solution calorimeter. This test consists of determining the heat of the solution of hydrated and the unhydrated cement by dissolving the samples in a mixture of acids. The difference between those two measured heat of solution values is the heat of hydration of cement. This test is normally used for cement paste and perfume after 7 and 28 days of hydration and the test results are comparable to the values obtained from the conduction calorimeter. The disadvantage of the test is that only small paste samples are used, not mortar or concrete samples.

Other Test Methods

Except for the four major types of calorimeter discussed above, the unthermostated heat conduction calorimeter can also be used to monitor the heat evolution rate. Wadsö (2004) developed a 14-channel unthermostated calorimeter based on the similar theory as the isothermal calorimeter. The performance of this calorimeter is governed by the ambient temperature stability and the heat capacity balance between each sample and its reference. Wadsö (2004) stated that the output can be used to quantify the retardation, detect cement-admixture incompatibility, and investigate the hydration disturbance.

Other than the calorimeter method, there are other simple tests used to test the sample temperature. These tests include coffee cup, dewar, bucket, and so on.

Coffee cup is an easy test to conduct with past samples. Together with the thermocouple, it gives the temperature of the test sample and reference sand. The temperature difference between the sample and reference sand and shape of the curves will be analyzed. It is a good method to check the compatibility of materials. And it works well for cement with SCM or admixtures.

The dewar test can be used to monitor temperature development with time for both past and mortar samples. The size of the sample depends on the size of the dewar. The quality of the insulation could vary for different dewars. The dewar test is easy and cheap. However, only the temperature can be obtained using this test. The environment conditions can influence the test results.

Lafarge Company uses the insulated bucket to test the temperature history of a concrete sample. A 4x8 mortar or concrete sample is used in the test. The sample is surrounded by the insulation materials. The temperature is recorded with time using the thermocouple.

Existing Temperature Datalogger and Sensors

Temperature sensors and datalogger are used to measure and record the sample temperature history. There also are many different types of temperature sensors and dataloggers available for monitoring cement and concrete heat evolution. With different temperature ranges and accuracies, their costs range from $20 to $1000. Models and computer programs for test data analysis are often developed for some specific heat evolution devices and sensors, which are generally sophisticated rather than simple and inexpensive. The detailed information in the temperature datalogger and sensors is in the appendix.

Existing Models for Heat of Hydration and Methods for Test Data Analyses

This section summarizes the models that are used by different researchers to predict heat of hydration, and the methods for concerting raw test data.

Heat of Hydration Model

Many mathematical models have been proposed to predict the heat of hydration or the degree of hydration, which is directly related to the heat liberated. Kishi and Maekawa (1994) and Maekawa et al. (1999) proposed a multi-component model for cement hydration containing fly ash and slag and considered the interdependency. The model is expressed as Equation 2.

$$Q_i = \gamma \beta_i \lambda \mu s_i Q_{i,T_0}(Q_i) \exp\left\{-\frac{E_i}{R}\left(\frac{1}{T} - \frac{1}{T_0}\right)\right\}, \qquad (2)$$

Where,

γ	=	Delaying effect of chemical admixture and fly ash in the initial hydration exothermic process
μ	=	Parameter expressing the effects of mineral compositions
s_i	=	Parameter expressing the fineness of OPC
β_i	=	Availability of free water, used to take the interdependence among component reactions into account.
λ	=	Amount of $Ca(OH)_2$, which governs the mutual interaction between Pozzolanic materials and cement

The reaction process of each mineral compound is expressed by the reference heat rate and the thermal activity. The reference heat rate gives the heat rate characteristic of the mineral at a specific constant temperature. The thermal activity describes the temperature dependency of each reaction. In this model the thermal activities, E/R, are different for each mineral compound

(Figure 15). However, the thermal activities of fly ash and slag are constant with no consideration of the different types of fly ash and slag.

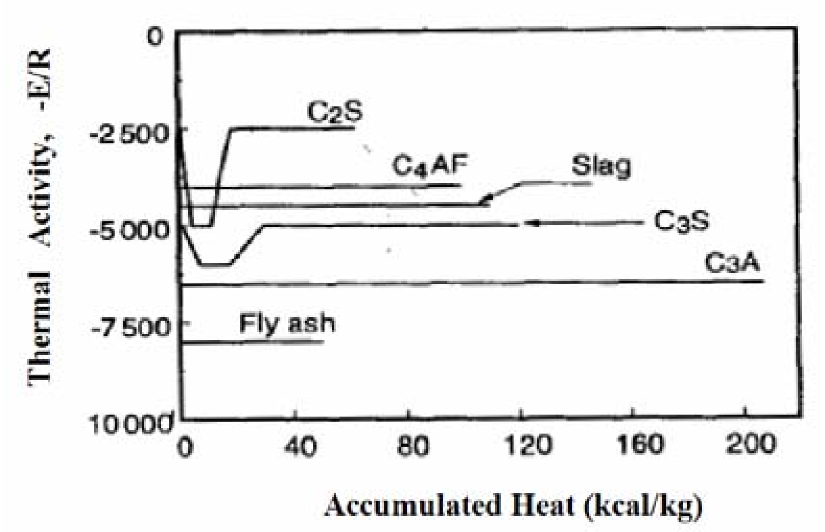

Figure 15. Thermal activity of each component (adapted from Maekawa et al. 1999)

Although cement hydration is a complex process, it is possible to treat the cement as a single material. Thus modeling the heat development characteristics of various types of cement is simplified. To achieve this, a thermal activity (E/R) value is used that represents the whole cement rather than each mineral compound as described in the multi-component model.

Suzuki et al. (1989) proposed the expression of the hydration process using the adiabatic temperature rise (Equation 4.2).

$$T_a(t) = T_\infty\left(1 - \exp\left(-r(t - t_0)^s\right)\right) \quad (3)$$

Where,

$T_a(t)$	=	Adiabatic temperature rise at t days after casting
T_∞	=	Ultimate temperature rise
r, s t_0	=	Experimental constant

This model represents heat evolution characteristics under thermally isolated conditions and can only be applied to the adiabatic situation.

In real situations, however, most concrete is not fully thermal isolated. To adequately capture the general properties of cement hydration, the temperature rise due to cement hydration should be taken into account. Several models considering thermal activity have been developed and are listed below.

1. Model Proposed by Byfors (1980), Oh et al. (2003), and Janasson (1984)

$$\alpha = \exp\left(-a_c \left(\ln\left(1 + \frac{t_{eq}}{b_c} \right) \right)^{-c_c} \right) \tag{4}$$

$$t_{eq} = \int_0^t \beta_T \beta_{W/C} \beta_W \, dt$$

Where,

α	=	Degree of hydration
a_c, b_c, c_c	=	Hydration parameters
t_{eq}	=	Equivalent age
β_T, $\beta_{W/C}$, β_W	=	Effect of temperature, water/cement (w/c) ratio, water distributions on rate of reaction

2. Model Proposed by De Schutter and Taerwe (1995)

$$q(\alpha, T) = q_{\max,20} \cdot f(\alpha) \cdot g(T)$$
$$g(\alpha) = \exp\left[\frac{E}{R} \left(\frac{1}{293} - \frac{1}{273 + T} \right) \right] \tag{5}$$
$$f(\alpha) = c \cdot [\sin(\alpha\pi)]^a \cdot \exp(-b\alpha)$$

Where,

q	=	Rate of heat generation
α	=	Degree of hydration
a,b,c	=	Hydration parameters
T	=	Temperature

3. Model Proposed by Freiesleben Hansen and Pedersen (1985) and Pane et al. (2002)

$$\alpha(t_{eq}) = \alpha_u \cdot \exp\left(-\left[\frac{\tau}{t_{eq}}\right]^{\beta}\right) \tag{6}$$

Where,

$\alpha(t_e)$	=	Degree of hydration at equivalent age of t_{eq}
α_u	=	Ultimate degree of reaction
τ	=	Hydration time parameter
β	=	Hydration slope parameters

Dabic et al. (2000) described a model that divides the hydration process into three separate processes, which are governed by nucleation and growth, boundary reactions, and diffusion respectively. The governing equations are listed in Equations 4.7 through 4.9. The parameters of KNG, n, K1, and KD were determined from the test data. This model well predicts the pure cement hydration, but for cement containing SCMs, deviations are shown in boundary reactions and diffusion-controlled periods.

- Nucleation and growth govern the hydration process.

$$\begin{cases} \alpha = 1 - \exp\left(-(K_{NG}t)^n\right) \\ \dfrac{d\alpha}{dt} = nK_{NG}^{n}t^{n-1}\exp\left(-(K_{NG}t)^n\right) \end{cases} \tag{7}$$

- Boundary reactions govern the hydration process.

$$\begin{cases} 1 - (1-\alpha)^{1/3} = k_1 t \\ \dfrac{d\alpha}{dt} = 3k_1(1-k_1 t)^2 \end{cases} \tag{8}$$

- Diffusion governs the hydration process.

$$\begin{cases} \left(1 - (1-\alpha)^{1/3}\right)^{1/2} = k_D t \\ \dfrac{d\alpha}{dt} = \dfrac{3}{2}k_D \dfrac{\left(1-(k_D t)^{1/2}\right)^2}{(k_D t)^{1/2}} \end{cases} \tag{9}$$

Where,

α	=	Degree of hydration
K_{NG}, n, K_1, and K_D	=	Hydration parameters
t	=	Time

Van Breugel (1995) developed a computer-based numerical model called HYMOSTRUC, based on microstructure development. This model can predict cement hydration in paste as a function of the particle size distribution, chemical composition, water/cement (w/c) ratio, and reaction temperature. Bentz et al. (1998) developed a three-dimensional (3-D) model for cement hydration and microstructure development. This model is able to predict hydration for cement containing silica fume. However, this model requires a two-dimensional image from SEM/X-ray analysis and the particle size distribution of the cement to establish a 3-D representation of the cement.

Methods for Calorimeter Test Data Analysis

The calorimeter test normally provides the temperature or heat liberated with time. However, most of the models listed above require equivalent age instead of the real time. Equation 10 shows the conversion from real time to equivalent age.

$$t_{eq} = \int_0^t \exp(-\frac{E_a}{R}(\frac{1}{273+T} - \frac{1}{273+T_{ref}}))dt \,, \qquad (10)$$

Where,

E_a	=	Activation energy
R	=	Universal gas constant, 8.314 J/(mol K)
T	=	Concrete temperature
T_{ref}	=	Reference temperature

The activation energy (E) is an important factor for equivalent calculation. The activation energy is regarded as the energy barrier to the chemical reaction. When the collision happens between the cement and water molecules, only molecules with kinetic energy higher than the activation energy can react. In the case of concrete, the activation energy is referred to as "apparent" because cement hydration involves several simultaneous and coupled chemical reactions. The characteristics of the individual mix, including cement composition, fineness, and mineral admixtures, influences the apparent activation energy (E_a). The w/c ratio may also have an effect on E_a. Carino (1984) initially found that the w/c ratio has little effect on E_a, except possibly for very low ratios. However, his later study with Tank (1992) showed that the E_a was dependent on the w/c ratio for several mix combinations. Kjellsen et al. (1993) and D'Aloia et al. (2002) also pointed out that E_a is dependent on the degree of hydration and it decreases at later age.

The apparent activation energy E_a could be determined in several ways. One of them is the strength test. This method is described in *ASTM C 1074 Standard Practice for Estimating Concrete Strength by the Maturity Method*. Concrete specimens are made and cured at several different temperatures. The strength age data is analyzed to obtain the activation energy. Recently calorimetric approaches have been proposed to determine E_a. It has been confirmed that Ea obtained based on heat of hydration is similar to E_a based on *ASTM C 1074*. Wirquin et al. (2002) shows that "calorimetric and mechanical means give very similar E_a values as the observed differences are close to 3 KJ/mol. The E_a determined by the method of "superposition" on the basis of the calorimetric results may legitimately be used to predict the strength of young concrete."

Some literature suggests that the activation energy should change with curing temperature. Below, RILEM (1998) recommends the activation energy for OPC and slag cement. The same value for slag cement is recommended regardless of the percentage of slag replacement.

For OPC

$$\frac{E_A}{R} = \left\{ \begin{array}{lll} 4000 & J/mol & for \quad T \geq 20^0 C \\ 4000 + 175(20 - T) & J/mol & for \quad T \geq 20^0 C \end{array} \right\} \tag{11}$$

For slag cement

$$\frac{E_A}{R} = 6000 \text{ J/mol} \tag{12}$$

Potential Uses of Calorimeter Test Results

The heat signature is directly related to strength gain and other various concrete properties, such as the setting time, sawing time and so on. Since the thermal heat signature of each batch of concrete is unique, it is possible to predict the change of the mixture. The possible uses of the heat signature curve are as follows:

- Predict the setting time
- Predict the proper curing time
- Predict the risk of thermal cracking
- Identify the sawing/finishing time
- Identify incompatibility problems
- Check cement characteristics
- Verify concrete mix proportion
- Select right mix for different environmental conditions
- Predict concrete strength

Other Related Research (Use of Dewar)

ISU has conducted some tests using different types of dewar devices. These tests were performed to evaluate (1) the difference between different types of dewar, (2) the effect of sample size, (3) curing environmental effect, and (4) effect of mineral admixtures.

Different Types of Dewar

Using two different types of dewar, glass and steel, a 2x4 mortar sample was prepared according to *ASTM C 305, Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.*

The temperature measurement began right after the mortar was poured into the plastic mold. Figure 16 shows the temperature of samples in the two different dewar and in the IQ drum. The sample in the IQ drum is used as a reference sample.

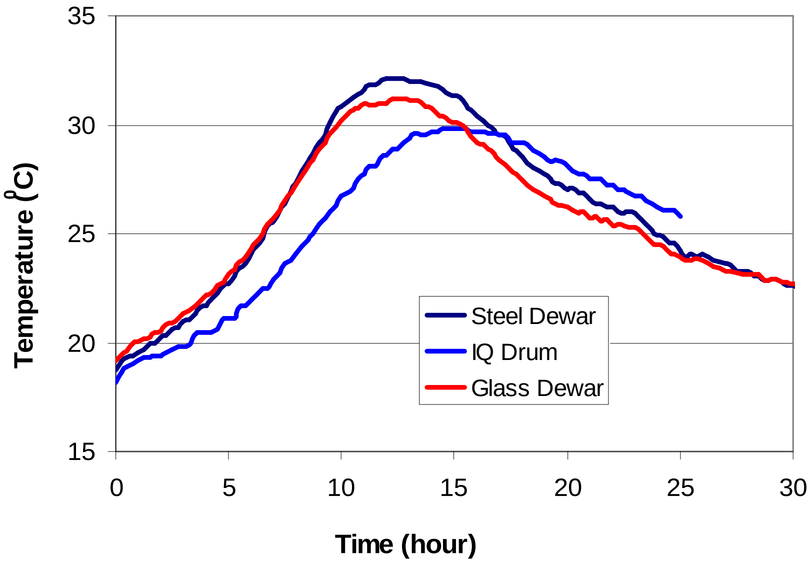

Figure 16. Temperature for samples in different dewar

Effect of Sample Size

Figure 17 shows the effect of the sample size. The temperature increases as the sample size increases. Also, the heat dissipates more slowly in the bigger sample than in the smaller sample. This indicates that the proper sample size should be selected according to different purposes.

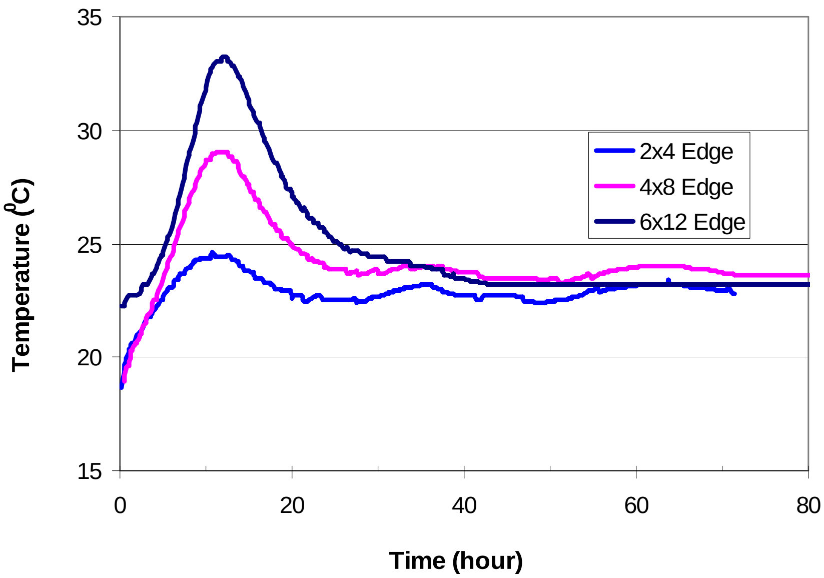

Figure 17. Effect of sample size

Effect of Curing Environment

Three samples were cast at the sample temperature. Immediately after the casting, samples were poured into the dewar and cured under three different conditions. The hot environment is about 31 °C, 20°C for the room condition, and 6 °C for the cold condition.

Figure 18 shows the temperature development for these three samples. It indicates that the peak temperature is reduced by the decreasing curing temperature. Under the cold temperature, there is peak. The position of the peak is also postponed by the curing temperature.

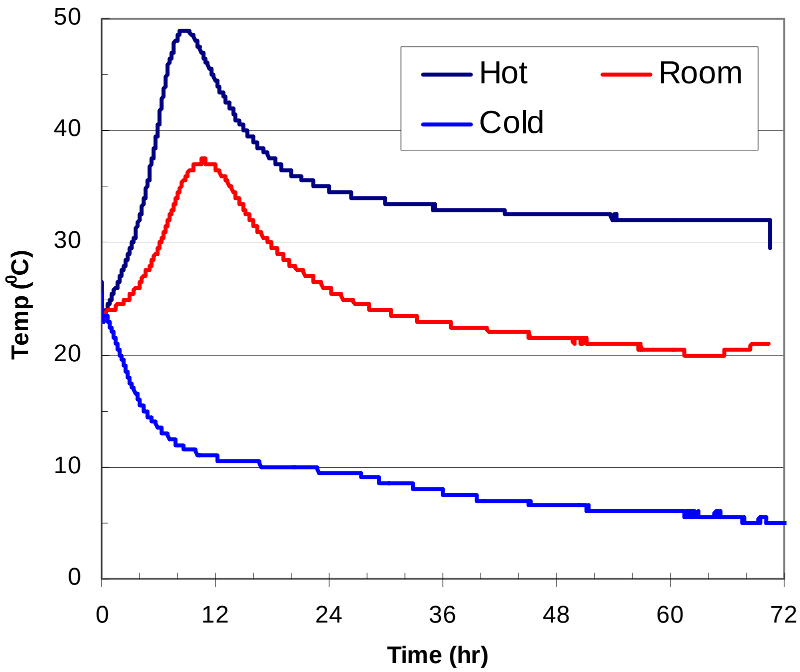

Figure 18. Effect of curing conditions

Effect of Mineral Admixtures

Tests were also performed to determine the effect of the fly ash and slag on cement hydration. Figure 19 and figure 20 show that both the slag and fly ash postponed the hydration peak. The delay caused by the fly ash is larger than that caused by the slag. As the mineral replacement increases, the peak temperature is reduced. However, the reduction is not proportional to the replacement level. This is due to the Pozzolanic reaction of the mineral admixtures.

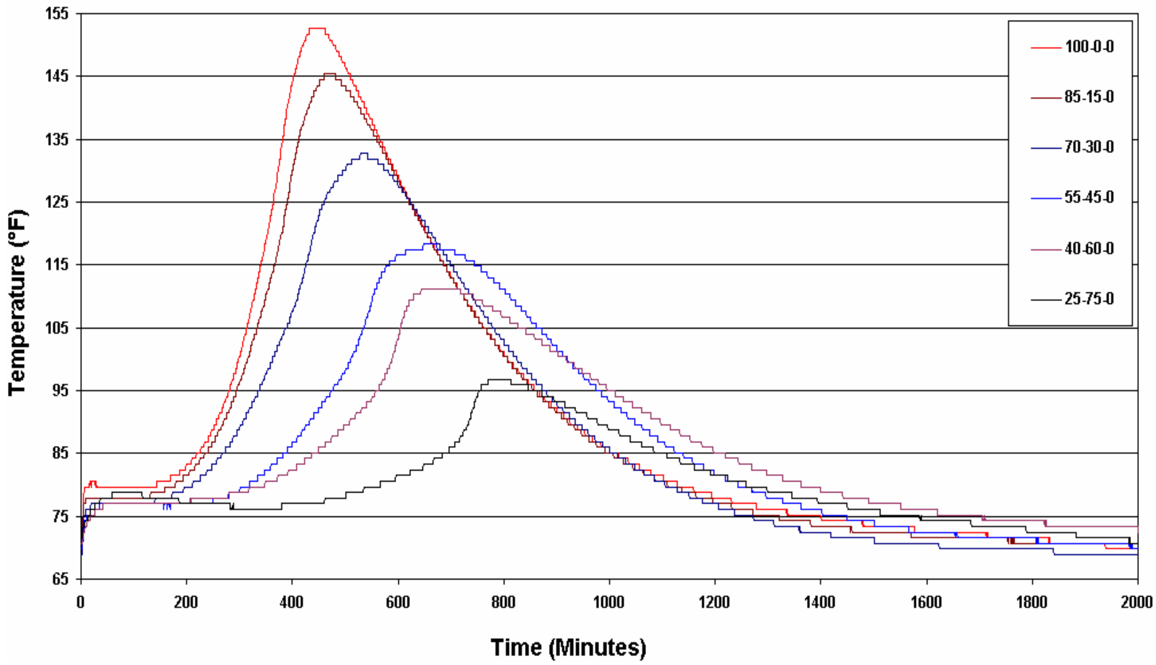

Figure 19. Effect of fly ash replacement

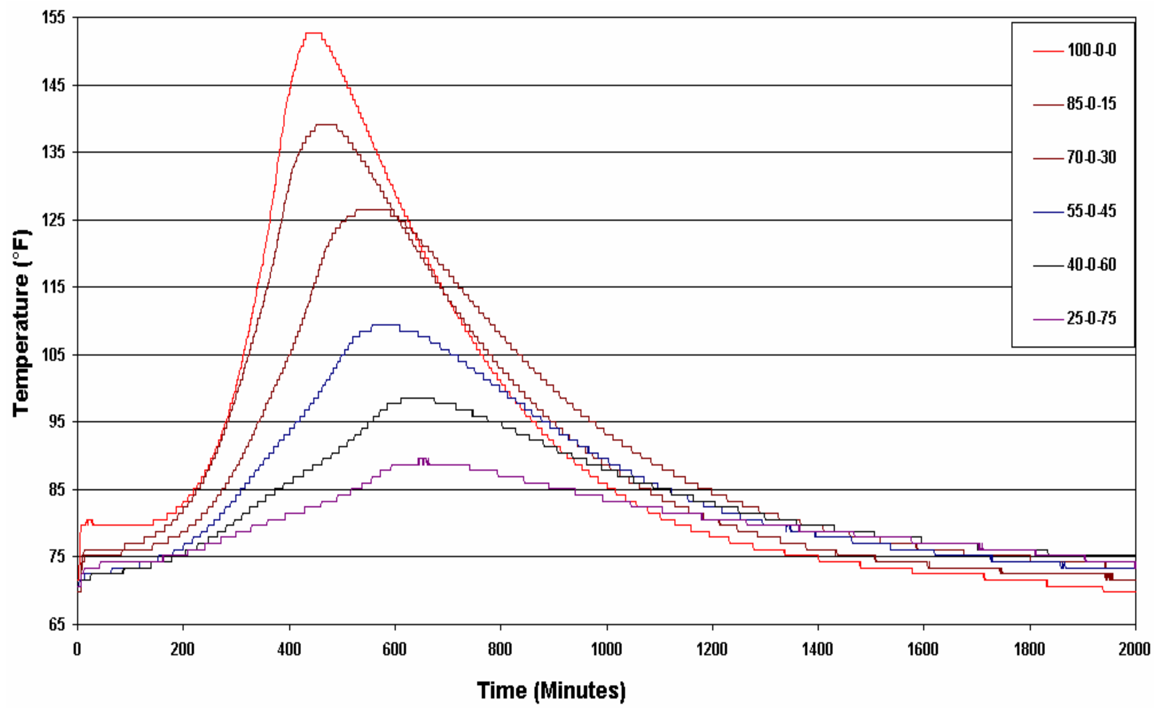

Figure 20. Effect of slag replacement

SUMMARY AND RECOMMENDATIONS

Hydration of the cementitious materials is a complicated process that contains a number of exothermic chemical reactions. The characteristics of the heat of hydration are unique for each concrete mix under each environmental condition. The hydration process is also directly influences concrete workability, setting behavior, strength gain rates, and pore structure development.

Hydration of the cementitious materials can be influenced by various factors. These factors include cement type, sulfate content, fineness, water/cement ratio, curing and initial temperature, supplemental cementitious materials, and chemical admixtures.

Different test methods are available for measuring heat of hydration, including the calorimeter method and other simple methods. There are four major calorimeters available: adiabatic, semi-adiabatic, isothermal, and solution calorimeter.

Many simple and inexpensive calorimetry tests have been used in a practical manner by the cement and concrete industry. Most of these tests are semi-adiabatic calorimetry tests, and some (such as the unthermostated heat conduction test) are semi-isothermal calorimetry tests. The identified simple, inexpensive devices for heat evolution tests include Dewar, coffee cup, and sprayed-foam basket. These test methods generally provide critical feedback within 12 to 48 hours. They have been used for investigating effects of SCM or chemical admixtures on hydration as well as compatibility of these materials. However, the accuracy and sensitivity of such simple tests are rarely reported.

There also are many different types of temperature sensors and dataloggers available for monitoring cement and concrete heat evolution. With different temperature ranges and accuracies, their costs range from $20 to $1000. Models and computer programs for test data analysis are often developed for some specific heat evolution devices and sensors, which are generally sophisticated rather than simple and inexpensive.

In addition to the maturity/strength prediction, concrete heat evolution test results can also be used for the following:

- Predicting concrete setting time
- Specifying curing period
- Estimating risk of thermal cracking
- Assessing pavement sawing/finishing time
- Characterizing cement features
- Identifying incompatibility of cementitious materials
- Verifying concrete mix proportions
- Selecting materials and/or mix designs for given environmental conditions

PROPOSED WORK PLAN FOR PHASE II

Research Objective and Scope

After reviewing the initial results from the Phase I study, the research team members had several discussions and identified the major gaps between the existing calorimeter tests and the needs of the pavement industry on calorimeter tests. The research team found that although various calorimeter tests have been conducted for assorted purposes and the potential uses of calorimeter tests are clear, there is no consensus on how to utilize the heat evolution curves to characterize concrete materials and how to effectively relate the characteristics of heat evolution curves to concrete pavement performance. The research team believes that the goal of the Phase II study is to close these gaps.

In phase II, the research team will conduct a more focused systematic study that combines test equipment and procedure development, heat evolution curve characterization, pavement performance prediction, and test/equipment specifications. The research team will target, but not be limited to, commercially available devices for the proposed lab study. The selected device is intended to be used to characterize cement properties. The research team will determine the applicability of the devices to the goals of this study. The hope and expectation is that these will meet the needs envisioned.

Given that the CP Tech Center's cooperative agreement with FHWA runs out in June of 2006, the research team proposes to have two parts (Parts A and B) in the Phase II study so that the Phase II–Part A can be completed by June 2006.

To further implement the Phase II research results, Phase III is also recommended to develop a much simpler, inexpensive calorimeter for field concrete.

Proposed Phase II Study

The whole Phase II study consists of five tasks: (1) conduct lab experiments using commercially available equipment to provide fundamental information on the establishment of criteria for calorimetry device evaluation and test procedure development, (2) establish heat evolution indexes to characterize concrete materials, (3) predict concrete performance using heat evolution indexes together with the HIPERPAV computer program, (4) develop a performance-based specification for calorimeter equipment and test procedures, and (5) conduct a preliminary study on the field application of the newly-developed calorimeter test methods and procedures. Tasks 1 and 2 will be completed in Phase II–Part A, and Tasks 3-5 will be accomplished in Phase II–Part B.

Phase II–Part A (Tasks 1 and 2)

Task 1

Task 1 is to conduct lab experiments to provide fundamental information on the establishment of criteria for calorimetry device evaluation and test procedure development.

Based on the results from the Phase I study, most currently available calorimetry equipment (such as an IQ drum) costs over $35,000. The research team will target, but not be limited to, commercially available devices for the proposed lab study. The research team believes that a device with a lower cost could provide the needed level of accuracy to meet the goals for its application to pavements. Up to two calorimetry devices will be selected. These devices shall be capable of characterizing major features of a concrete heat evolution curve with a desirable accuracy and a reasonable price (affordable by most State DOTs). Currently, an unthermostated heat conduction test device (a semi-isothermal calorimeter manufactured by Thermometric Inc.), TAM Air, is already commercially available at a cost of approximate $37,000, and it has been used by W. R. Grace for characterization of cement properties and study of admixture effects on pastes. Another device from the same manufacturer (Thermometric Inc.) will be coming on the market this fall, which will have similar functions and a considerably lower price (approximately $8,000). This device, together with others (such as IQdrum), will be considered in the proposed study.

During the course of the device evaluation and test procedure development, a range of paste and/or mortar mixtures will be used. (Mortars sieved from concrete mixtures may also be used.) The following variables will be considered:

1. Sample Materials and Proportions
   Paste and/or mortar samples with and without different levels of fly ash and GGBFS replacement, cement content, and different amount and types of water reducing agent will be tested. The mixes with different types of cements and water/cement ratio may also be studied.
2. Sample Size
   The size for the cement sample will likely be no larger than the 2" x 4". The mortar and concrete samples will have bigger sizes, 3" x 6" for mortar.
3. Environmental and Placement Temperatures
   An ambient temperature of 70°F will be used for all trial equipment, since it is similar to most concrete laboratories. However, field-readiness testing on select equipment will include the use of ambient temperatures ranging from less than 50°F to 90°F or greater. It is expected that initial concrete temperatures will also vary. Samples with different initial temperature will be cast.

The test results from different calorimeter measurements will be compared. The setting time, premature stiffening, and strength of the samples will also be tested, and the results will be compared with characteristics of the heat evolution curve. It is expected that these data analyses will provide the research team with insight into the validity and accuracy of different calorimeter equipment and test methods for various paste and mortar mixtures. This information will assist in development of the acceptable criteria for equipment selection and test procedures.

<u>Task 2</u>

Task 2 is to establish heat evolution indexes to characterize concrete materials.

After a calorimeter measurement, it is very important for engineers to interpret the meaning of the heat evolution curve correctly and effectively. This task is to establish heat evolution indexes that will help engineers to interpret the calorimeter test results. These heat evolution indexes can also help researchers and engineers to define or describe a heat evolution curve. The characteristics of a heat evolution curve defined by these indexes can be further used for concrete quality control.

As shown in Figure 21, at least five to six parameters (t_0, T_{max}, t_{max}, S_1, S_2, and A) are needed to define/describe a specific concrete heat evolution curve. These parameters are closely associated with concrete properties and performance. Models and software will be required for converting the raw test data into relevant test metrics, or heat evolution indexes. The development effort will advance the test procedure to a beta test stage. The software will be developed so that the major parameters that characterize the features of the concrete mixture are presented in an easily readable format in the test report. Potential parameters will likely include the peak temperature, slope of the initial heat curve, and total heat librated from concrete, along with the rate of reaction. It is anticipated that the software will be an integral part of the overall system, as it will be able to take the phenomenological results of the test device and convert them to more fundamental measures of the heat of hydration. The result will be a procedure that is both repeatable and reproducible.

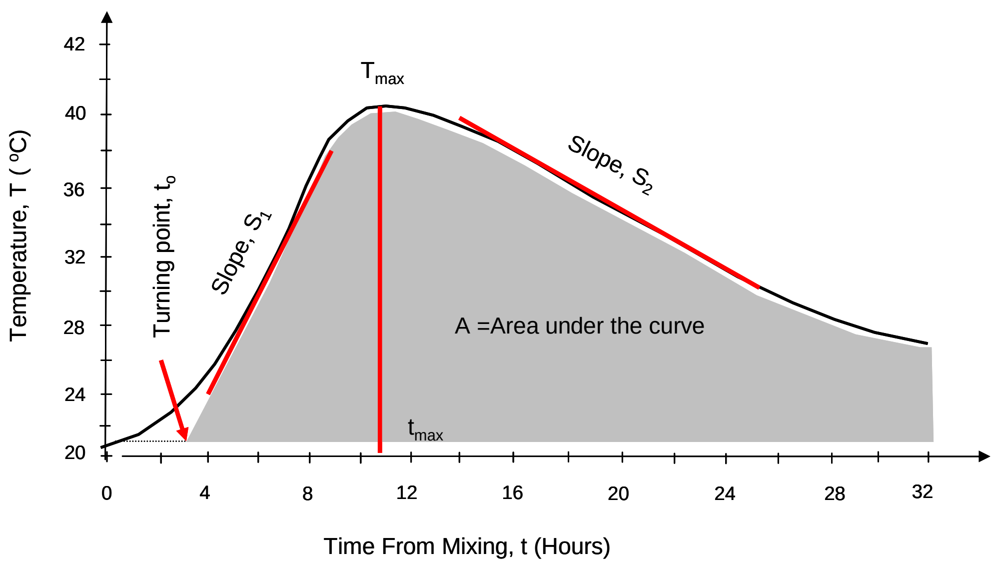

Figure 21. Characterization of a typical heat evolution curve

Phase II–Part B (Tasks 3, 4, and 5)

Task 3

Task 3 is to predict concrete performance using heat evolution indexes and HIPERPAV software.

This task is to utilize the calorimeter test results to predict concrete pavement properties and/or performance. Recently, the Transtec Group Inc. and FHWA completed a research project focused on modeling the early-age development of concrete strength and stress that result from moisture and temperature changes within the pavement. A comprehensive software package, HIPERPAV II, has been developed during this effort. HIPERPAV II can be used for concrete quality control, optimization of pavement designs, prediction of pavement performance, and help contractors in managing the temperature of concrete based on the concrete mix designs and specific climate and project conditions.

Currently, the HIPERPAV program uses predicted heat evolution results based on cement characteristics and concrete mix design. Heat evolution information is a fundamental input to HIPERPAV for the prediction of the pavement strength and stresses during the early age. The proposed task is to use the calorimeter test results (or heat evolution indexes) as input data for the HIPERPAV program analysis, thus improving reliability of the HIPERPAV analysis. The research team envisions that a success of this task will facilitate applications of both the HIPERPAV program and calorimeter tests in the concrete pavement industry.

During this task, the project team will develop sensitivity analyses of heat evolution curves to determine the changes in pavement performance. This information will be needed in determining the minimum precision and accuracy that calorimetry devices will have to meet for use in paving applications. In turn, this information will be used to establish minimum requirements for development of a field calorimetry device.

Task 4

Task 4 is to develop performance-based specification for calorimeter equipment and test procedures.

The research team will work closely with the TWG in the development of the criteria for accepting the test results. Also, the information collected in Phase I and Phase II Task 1 will be considered in the development of criteria. Along with a final report, the research team will draft a specification for calorimeter equipment selection and test procedures based on the AASHTO/ASTM specification format. In order to not infringe on commercial interests, the specification will be written in a performance based approach focusing on results tolerance and bias rather than on a rigid procedure.

Task 5

Task 5 is to conduct a preliminary study on the field application of the newly-developed calorimeter tests.

After achieving initial research results from Tasks 1-4, the research team will conduct up to three calorimeter tests for field concrete following the newly-developed test procedures. These field tests will be incorporated with the ISU Concrete Mobile Lab activities for the Materials and Construction Optimization project. The test results will be used for refining the performance-based specification developed in Task 4. The team will also work with the TWG to determine the best course of action to further evaluate and implement the results of this project.

In phase II, the project TWG will continue assisting in steering the direction of the research, providing input and critical viewpoints on test devices and systems, criteria, and guideline development. They will also participate in the evaluation of the research products at various interim stages.

Research Period and Budget

Table 3. Projected research period and budget requirements

	Project Dates	Required Amount
Phase II–Part A (Tasks 1-2)	October 1, 2005 – June 30, 2006	$72,000 (including $8,000 for device)
Phase II–Part B (Tasks 3-5)	April 1, 2006 – September 30, 2007	$90,000

Anticipated Results

The following products will be submitted as indicated:

1. A set of criteria for calorimeter equipment selection and development
2. Test procedures for evaluating the heat of hydration of cement, mortar, and concrete mixtures using a calorimetry test technique
3. A set of indexes for characterization of heat evolution curves
4. Accompanying models and software that are capable of converting the raw test data into easily understood test results.
5. A draft of an AASHTO specification for the recommended test method
6. A final report that documents the results of the entire study

Implementation

Implementation of the project results will be conducted through field applications of the newly developed test device and procedure, and will occur in Iowa and throughout the Midwest. The device will be field tested and demonstrated through the CP Tech Center's mobile concrete laboratory. The research results will be disseminated through presentations at technical meetings (such as TRB, ACI, and Midwest Concrete Consortium [MC2] meetings), and publications of the report and papers.

Level of Importance to Overall Goals

It is believed that the research results will benefit all agencies related to concrete materials and construction, especially the concrete paving industry. The test results and specifications will allow identification of changes within the concrete mixture due to material changes, thus adding a significant tool for quality control of concrete mixtures

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APPENDIX A: HEAT EVOLUTION TECHNICAL WORKING PANEL (TWP) MEETING MINUTES

HEAT EVOLUTION
TECHNICAL WORKING PANEL (TWP)
MEETING

1 October 2004
Des Moines, Iowa

MEETING MINUTES

Attendees

TWP Members:	***Research Team:***
Gary Knight (GK) – Holcim	Jim Grove (JG)
Paul Sandberg (PS) – WR Grace	*Kejin Wang (KW)*
Peter Taylor (PT) – CTL	*Zhi Ge (ZG)*
Anton Schindler (AS) – Auburn University	*Ted Ferragut (TF) – TDC Partners, Ltd*
Todd Hanson (TH) – Iowa DOT	*Robert Otto Rasmussen (RR), The Transtec Group*
Leif Wathne (LW) – FHWA	*J. Mauricio Ruiz (MR), The Transtec Group*

Project Overview (JG)

- JG welcomed every body to the meeting and started introductions.
- JG provided an introduction of the project. He explained that we don't already have a plan that the TWG is validating – the plan will be generated by all of us. JG hopes that with the experience of all at the table we will get a good idea of what we should develop under this study.
- JG provided a brief description of the Materials and Construction Optimization (MCO) pool fund project that ISU is currently undertaking. He mentioned they have a small and a large i-Q Drum in the trailer.
- A problem was found when the trailer is in a place with no cell phone service – they couldn't connect to the Internet to analyze the i-Q Drum data. This validated the fact that the i-Q Drum is not a field test.
- There is a way to store the i-Q Drum data and later send it over the Internet for processing.
- A test is needed that can be done out in the field that gives you information about your mix.
- PT sees this maybe as a process control, or maybe for record keeping?
- JG says that it could possibly be used both ways.
- How do we assure that we use the right mix for the right application?
- A good example is supplementary cementitious materials (SCM) – in late season placement, the mixes with SCM can cause problems.
- Can we characterize mixes in a way that we can select the right mix for the weather conditions?
- Slump can really only tell you changes – not an absolute. GK says that this has always been the case – it was not developed for a measure of workability, but instead consistency.
- TH said HIPERPAV could use calorimetry data as an input.

- JG Proposes characterizing mixes with a **"Heat Evolution Number (HEN)"**.
- Funding limitations because of the highway reauthorization made it necessary to cut this project into two phases. This first phase is to develop a comprehensive plan. The second phase will be to develop the specification and prototype equipment.

Implementation thoughts (TF):

What do we want at the end of Phase I?
What do we want at the end of Phase II (5 years)?

- Piece of Test Equipment.
- Characterizing mixtures at the mix design level (**At the lab**).
- We also need a Procedure.
- Data Interpretation.
- Field Equipment.
    - Characterizing mixtures for quality control: Go/no-go for delivery.
    - **At point of placement.**
    - Wants the "answer" in 15 minutes (desirable). 24 hr may be more realistic.
    - May not be a rejection test, may be a warning to use different construction procedures to make it work.
    - **At the plant.**
- What does it look like?
    - Has to fit in the mobile lab trailer: length, width, depth: 2-3'.
    - Mounted in a truck, driven to the site.
    - Concrete sample – allowing multiple samples.
    - Needs to be portable – to be picked up and put down.
    - Needs to be "on the side of the road" for maybe 24 hours.
    - Similar to maturity? Need to consider the same concerns as maturity devices (getting run over, stolen, etc.).
- If you are going to reject – need to test at the plant to give the lead time to possibly reject at the site.
- What is the technician level that should be considered (qualifications)?
    - DOT inspector.
    - "Typical" lab personnel.
    - Similar training requirements to maturity.
- Are we limiting ourselves to paving concrete? What about structural concrete – especially mass concrete?
- Can we really reject concrete due to heat of hydration (HOH)?
- LW says that in some cases, validation of the maturity curve is only done periodically… this assumes a gradual change in the mix. In reality, changes can occur quickly and dramatically.
- In a field production mode, no matter the duration of the test, the concrete will likely be on the grade. This test might allow for curing measures to be modified. Makes it difficult to see this in a quality control mode.
- Size, ruggedness, and cost are all important variables.

- The ruggedness will likely increase the cost – particularly important if we leave "on the side of the road".
- Making a lab test, the ruggedness is not as critical.
- For cost comparison: AVA is $25k.
- Test results in the lab can be used for mix design.
- At the batch plant, the interpretation may be different.
- At the plant, grab a concrete sample, and if you can get an answer in less than 15 minutes, you can call off the placement.
- Beginning to sound expensive.
- Consistency (changes) or a go/no-go?
- Maybe to address batch-to-batch changes in cement shipments. Test mixtures using samples of the loads with pre-mixed aggregates, and see how the results change.
- What is the ruggedness in this case? Maybe not as much as in the field (on the road).
- Might be nice to have a product that can be used at all three locations – maybe have additional protection for the field version.
- Location and price that the market will bear:
    - $6,000-8,000.
    - < $100 (at the site).
    - At the site: $2,000; In the lab: $8,000.
    - $100 disposable to $8,000 reusable.
    - For simple go/no-go: $100.
    - Depends on what the product will do.
    - Value the device based on the output.
    - Reusable base device + disposable components.
- There should be availability by multiple vendors.
- Okay to have multiple types of equipment at this phase – the next phase will likely require that we pick one or two or three of these.
- We need to make sure that the technical concept supports what we are developing.

Introduction on technical issues on calorimetry tests (KW).

- Outlined the benefits of heat evolution testing.
- Presented some applications of heat evolution testing on concrete pavements.
- Outlined the research objective for this project and the project timeline.
- Outlined major considerations in the test development.
- Presented some of the calorimetry tests currently available.

Experience with a "coffee cup" test calorimeter (GK).

- He uses a reference sand sample and other samples with different combinations and proportions of cement, SCM, and admixtures.
- He can determine set time with a change in the slope of the heat curve.
- He some times runs duplicates. They usually come pretty close. He recommends running duplicates.

- These tests can be done as part of a cement shipping requirement.
- This will work better when doing combinations of SCM or admixtures with cement. Or change of cement supplier.
- The temperatures shown on the hydration curves is meaningless, it is more the temperature difference and shape of the curves that make the results relevant.
- You can test set retarders. Some perform very uniform with only a retardation effect and not much change in hydration curve. Others have a significant effect on the hydration curve besides retardation.
- A temperature of 10 degrees made a compatible mix into an incompatible one.
- Good method to look at compatibility of materials.
- Can be used to test overdose of admixture.
- At different temperatures, the effects may be totally different.
- All are done with paste samples.
- When you do this with concrete the tests turn too noisy due to the presence of aggregates.

Texas DOT hydration study (AS).

- Cautions the use of the Qdrum as the gold test.
- Qdrum does not account for temperature sensitivity.
- Need to update that accordingly.
- Need to have knowledge of your thermal conductivity of the materials.
- Mayor errors may be obtained with imprecision in assumptions (e.g. slag aggregate properties). From a few degrees to 15 ºF or more.
- They have tested more than 400 different admixtures to redefine the hydration models.
- Uses a three-parameter model for hydration. The model has worked quite well so far.
- You need to account for thermal sensitivity. RILEM has recommended some methods for this. Activation energy is one. There may be some problems with this approach.
- Using isothermal calorimetry testing to see if activation energy testing can be improved.

CTL experimental calorimetry study (PT).

- CTL has two isothermal calorimeters:
- Wexham, 2 cell, 72 hr minimum test duration. Variable temperature range.
- Toni-Teknik, 1 cell plus reference. Single temperature.
- Working on a project for NCHRP.
- Tests of different SCM. Testing 900 mixtures… in progress. Haven't done much interpretation yet.
- Working also on an incompatibility project. Calorimetry is very good in identifying incompatibilities.
- Interactions with admixtures may be very sensitive.
- They are doing tests on concrete with simple testing equipment (4" x 8" cylinders).
- They monitor the rate of cooling to calibrate the hardware for heat loss.
- They are also working in the field monitoring temperatures. Second derivative of the shape of the temp curve is correlating very well with set time.

General discussion

- LW – Instead of taking a sample and see if we can duplicate results in the field, why don't we use models to predict effects of environment and backcalculate a signature from in situ conditions.
- We need to have a good handle of how that concrete will behave.
- This is what we are trying to determine to see if we can capture all the variables that affect heat development in the field.
- There are so many variables that can affect this in the field. If we use a semi-adiabatic calorimeter that take away some of these parameters may be a better start.

Cement-admixture compatibility studies by isothermal calorimetry (PS).

- 99% of all interaction problems occur with materials that themselves comply with existing standards.
- Different admixtures under field conditions may perform totally different.
- Isothermal calorimetry can capture heat reaction at the start of hydration.
- Testing for effect of overdose will indicate how close a mixture is to being out of balance. How much overdose can be afforded?
- Can also test delay of addition. Even a few seconds allows the aluminate to hydrate initially without admixture.
- Field calorimeter developed, currently used in California.
- Ready mix people doesn't go through these tests. At least not in a regular basis.
- Cement manufacturers do.

General Discussion

- Iowa some time ago reduced the limit of sulfate content in cement, this was leading to other problems with admixture compatibility.
- We need a way to characterize these curves better.
- There should be an ASTM for people to use it.
- Why we don't use it?
- PT sees a future for selling services on the use of calorimeters.
- We need a base line curve. We may depart from an isothermal test at different temperatures or from an adiabatic one.

Instrumentation Ideas (RR)

Why are we doing this?

- *Identify changes in the concrete mixture.*
    - *What is significant enough to be considered a "change"?*
    - *What do you (as the inspector) do if a "change" is found?*

    - *Need a clear table/flowchart of what the change might be caused by, and what the inspector can do or look for.*
- Need to know the standard deviation in the test for the same materials.
- How different is the same material in the test?
- How different is the "same" material within the production?
- Sensitivity of the mixture – small changes to the test conditions may affect the results.
- Need to know what caused the changes. Is it difference in cement, admixtures, climate, batching sequence, time of mixing, weight of the sample is critical, delay from batching to casting?
- Modeling can help you determine difference in setting, and other properties. A change in degree of hydration can translate into a change in PCC properties.
- What caused that change? E.g., differences in test temperature? Can we normalize these?
- The bigger the sample, the more heat will be generated. May be able to correct for the weight of the sample.
- The delay from batching to casting lab specimen to insertion into the device.
- The difference in time from the time the PCC is on the grade to when the test begins.
- To determine what is a significant "change" might just take experience on the paving operation.
- A good example is slump… only experienced operators will know what is significant.
- Maybe need to relate the HOH to setting, and determine the sensitivity from that.
- By the time you get the curve, the set time will have happened anyway.
- Tie HOH to performance and determine sensitivity based on that.
- Specify what to do when the "flag" is raised from a significant change.
- If pavement has already been placed, not sure of the value added of doing this in the field.
- This test may be of value in the lab for compatibility, for example. Good to have a better and cheaper way of doing things now.
- Could a specification be developed that says you have to be in the "green zone" x% of the time.
- In order for the specification to work, you not only need to link HOH to the performance, but HOH to the materials changes.
- You also need to make sure that the change is not due to noise or a bad batch.
- This test is not an accelerated test, so the feedback is not instant. We may see problems after they happen, may be too late.
- Can HOH be used to track mix changes like strength tracking?
- Doesn't see this being used to reject concrete.
- Instead, look at this like HIPERPAV to flag when process changes need to be made.
- This test is providing guidance… a tool that helps in decision-making.
- An advisory test is the best use of this.
- This can be used to validate maturity curve in the field.
- The RILEM committee's objective was to look at early-age cracking.
- This kind of testing is ideal for looking at this in advance of placement.
- The market may be limited if it is just an advisory tool.

- HIPERPAV does not have a large distribution for similar reasons.
- Warranties may open the door to use this. As it has happened with HIPERPAV and its use in Michigan.
- Repairs "shall be made by the contractor at no cost to the DOT"… this changed how things were done.
- Current specification for mix deisgn / mix changes may be factor in the use of this devices.
- Find a window for sawing is a potential use – this may be a more rigorous way of doing this – in real time.
- Sawing usually happens on the "low end" of the maturity curve – temperature may be a better indicator.
- Maybe need to see how the device is going to be used before we build guidance on mix changes.
- What we do will depened on where the test is run, and what control that party has.
- There should also be guidance on retesting if a "bad" number comes up.

➢ *Inputs for HIPERPAV*
  - *Maybe need a HIPERPAV "light" for only strength gain predictions/potential – blend this with maturity specifications.*
- Some states have a bridge spec for settlement cracking over piers. Want setting to occur to minimize this cracking from occurring. May be used to obtain simultaneous setting for bridges by changing admixture dose. Retarder dose has to change as placement progresses.
- Mass concrete – use this for determining initial concrete temperature or need for cooling.
- In prestressed, PCI guidelines have a maximum concrete temperature.
- Delayed ettringite formation might drive some maximum concrete temperatures – mostly in mass concrete, but some fast-track concrete reached these critical temperatures.

➢ *Catalog mixes*
  - *Planning for future jobs.*
  - *Effect of SCM, chemical admixtures, etc.*
  - *Mix optimization.*
- Define "seasonal" mixes.
- Identify "HEN" numbers that define when mixtures could be used. A specification could be used to say what mixtures could be used depending on the environmental conditions.
- Maybe define a ratio of numbers between peak and set temperatures – something to define an index.
- Low point, high point, slope, offsets… any/all of these could be test metrics.

Targets we should aim for:

➢ *Cost*
  - *"Fixed" equipment (e.g., Pocket PC).*
  - *Reusable equipment (e.g., temperature sensors, cylinder molds).*
  - *Reasonable life-cycle cost of equipment considering wear & tear.*

- A lab-based device may need to be more expensive due to increased accuracy and precision.
- Field-based tests could possibly be calibrated off of the lab-based device.
- Industrial floor market – people will pay if they can increase the quality.
- $2,000-$5,000 reusable (for the lab version).
- The lab one may not be needed since it is available now. We may still use some off the shelf with some modifications.
- Who buys it? Some states require the contractors to buy them.

- *Ruggedability*
    - *Weak points – can they be hardened? (e.g., Pocket PC).*
    - *Wires strengthened?*
    - *Sensors protected?*
- Lab version doesn't necessarily need to be as rugged.
- Bull-dozer proof? Kick-proof? Pickup-proof?
- Rain, freezing temps?
- The field device – will it really be left in the field, or in the trailer?
- Probably at the batch plant or at a field office.
- Indoors to help with temperature control, but may be "thrown" in a pickup for transport.

- *Ease of use*
    - *Training requirements.*
    - *Length and complexity of instructions.*
    - *Prerequisite knowledge of concrete.*
    - *Anticipated operator errors and impact to test results.*
    - *Weight of equipment.*
    - *Bulkiness (size) of equipment.*
    - *Number of parts.*
    - *Ability to replace parts in the field.*
    - *Location requirements (e.g., side of road, lab, etc.) – look to match the procedures of this test with others like strength beams.*
- Need to look at maximum weights for OSHA.
- 40 lbs?
- 2-3' "cube".
- Would be better with hinges/collapsible.
- Maximum size – 5 gal bucket.
- Possibly a little bigger – even as big as a 55-gal drum.
- How many samples will it run? If more samples, we will need a larger device.

- *Accuracy and precision – repeatability and reproducibility*
    - *As low as possible (but how low before the test is too "quick and dirty")*
    - *What should we use as the "golden" (standard) values to gage the device on?*
- Will depend on the lab vs. the field device.
- Tied to the repeatability of the test.
- With pavements not being the highest risk structures, the accuracy may not be as critical.

- Maybe isothermal calorimetry as the "golden standard".
- Might be better off calibrating to fully-adiabatic conditions. Large specimen calibration (4x4x4') is one possibility. Ensure that calibration is done at a temperature that is not too elevated. For example, lower cement factor for calibration test to reach low temp.
- Isothermal might pose problems with temperature sensitivities that a (semi-)adiabatic test would encounter.

- *Calibration requirements.*
  - *How often (1 year +).*
  - *Cost (low).*
  - *Who (state DOT central lab or manufacturer?).*
  - *How (easily – maybe using water or a heated metal specimen).*
- Use of the device will drive the need for calibration.
- Watch out for wear and tear – since it might drive the need for more frequent calibration.
- We are calibrating both temperature sensors and heat loss.
- European devices maybe can be used for calibration.

- *Spur competition among device makers*
  - *Develop a functional specification! Not prescriptive.*
  - *See the "Nordic" spec cited by RILEM 119-TCE.*
- Functional specification: Define required limits. Test results need to fall within certain tolerances. A 30-page spec can be reduced to 2-3 pages this way.
- We should explore a functional spec as an option.
- Find how sensitive the results are to a fixed activation energy value. Problematic to determine activation energy. If not needed better.

Project tasks (in no particular order):

- *Literature search and collection of references*
  - *Reports.*
  - *Specifications (ASTM, CEN, RILEM).*
  - *Manufacturer literature (and costs) including Dewar.*
  - *Start with RILEM 119-TCE references.*
  - *Patents.*
  - *Translation services needed for foreign specs?*

- *Contact 119-TCE Representative (Chair preferred – Springenschmid).*
  - *Ask if other reports of 119-TCE had been published?*
  - *Was there any follow-on work after the TC had concluded?*
  - *What impacts were reported to Euro practices/industry as a result of this TC?*
  - *Are the letter codes (A,B,C…) cited in the TC report public? In other words, what devices correspond to what codes in the round robin test results?*
  - *Is the semi-adiabatic to adiabatic correction method published in the Technical Recommendation the same as the "Grube" method? Is there more information available beyond "personal communication" that was cited as a ref?*
  - *Have there been intellectual property (patent) challenges in Europe regarding these devices?*

- *Sensitivity of maturity equations, thermal stress calculations, and HIPERPAV.*
    - *Incremental changes in max. adiabatic temperature and shape parameters as well as activation energy.*
    - *Compare to incremental changes in cement content and other variables.*
    - *Sensitivity to strength at various ages.*
    - *Sensitivity to stress (peak and ratio of stress-strength).*
    - *Simulate a semi-adiabatic test to determine sensitivity to changes in k and c_p of concrete (since these will need to be assumed).*
- The temperature of the test will affect the test results. How we use the device will affect the selection of this.

- *Investigate equipment options*
    - *Temperature sensors.*
        - *Cost.*
        - *Availability.*
        - *Precision.*
        - *Accuracy.*
        - *Repeatability.*
        - *What sensitivity (precision) do we really need since this is often tied to cost?*

    - *Insulation*
        - *Polyurethane?*
        - *Polystyrene (Styrofoam)?*
        - *Others? (ones that are cheap, uniform, and have a high insulation coefficient – the better the insulation, the more accelerated the test (that is good).*
        - *Talk to thermodynamics professor or Dupont (industry) or NASA.*
        - *Consider cost, availability, safety, practicality, consistency.*
- Be careful of higher temperatures since some admixtures will react differently.
- Need to stay within the temperature range expected of the application (in-situ pavement).
- Design the insulation of the test to represent the "average" concrete pavement.
- Be careful of going too far with adiabatic conditions – this is too hot.
- Q-drum insulation is very similar to the concrete pavements.
- New house insulation.
- Consider balsa wood.

    - *Computer platform*
        - *PC (laptop)?*
        - *Pocket PC?*
        - *1-wire devices and their interface? (iButtons and thermocouples for sure… but others?)*
- Nice if it could log its own data, and download. Not permanently connected.
- Generic computer – PC.
- More contractors would have laptops.

- Need to see who is using Pocket PC vs laptop.
- Power requirements for the test device – AC or batteries.
- AC could be shut down in the trailer at nights (generator stopped) – may need battery (at least a backup).
- Indicator of test progress / start.

➢ *Equipment calibration*
  - *Distilled water "specimen" (since thermodynamic properties are known).*
  - *Hardened concrete specimen (oven heated).*
  - *Consider the fact that ambient temperatures during calibration will be different than in the field… a correction will likely be needed when the test is run in the field for the effect of ambient temperature.*

➢ *Test procedure*
  - *Specimen size – ideally 4x8 cylinder, but maybe larger for larger aggregate?*
  - *Don't wet sieve mix! Just adds an extra step, plus it may make it more difficult to use the results.*
- Should be able to do both, mortar and concrete.
- Leave space for a 4" x 8" concrete sample – or have a sleeve to put a smaller mortar sample in it.

  - *In the field, do we need to consider the delay between time of batching and time of placement?*
- Yes

  - *Consolidation method for sample (tamping, vibration, etc).*
- Sample must be weighed – paste, mortar, or concrete.

  - *Temperature of the PCC vs. temperature of the (semi-)adiabatic chamber. Should be the same (or very close), but how?*
- Need for account for it. Mathematical corrections.
- Reference materials (like the sand in the coffee-cup test).

  - *Specific heat of chamber – criteria set in the RILEM spec.*
  - *Need to correct for ambient temperatures.*
  - *Keep shaded from sun (so we don't need to make corrections).*
  - *Keep out of wind (so we don't need to make corrections).*
  - *How is calibration "correction" factor included?*
  - *Is a parallel "calibration" specimen needed? Try to avoid if possible – probably not practical.*

Other thoughts and notes:

➢ *What about the initial spike in temperature upon initial mixing – from C4AF – do we need to account for that somehow?*
- Not as big of a problem in concrete.
- Cast the specimens quickly.

- *Maybe conduct a round robin in Phase II – modeled after RILEM 119-TCE maybe?*
- *Maybe even an informal round robin in Phase I with existing devices at ISU and under control of the TWG?*
- Round robin would provide more of a national validation.
- Get other labs involved after the TWG has tested it.

- *Look for more information on the "Grube" method cited in the RILEM TC for converting semi-adiabatic to adiabatic.*
- *Consider recent findings of different Activation Energies for maturity and heat development… consider this before fully accepting RILEM calculation methods.*

(Meeting Adjourned)

APPENDIX B: TRANSTEC GRUOUP TECHNICAL MEMORANDUM

THE TRANSTEC GROUP

TECHNICAL MEMORANDUM

To: Project Staff
From: J. Mauricio Ruiz
Re: Field Calorimetry Study – Consensus Thoughts (DRAFT)

Memo No. 204018-2
Date: 24 January 2005

After our very productive meeting on October 1st, the project team was left with a number of ideas that will help in shaping the development of the field calorimetry test. There were however varying opinions on the specific problem we are trying to address and the way it should be addressed. This document aims at summarizing the thoughts expressed at the TWP meeting on this specific issue. It is believed that making this concept clear is important before continuing with the details of test development. It is important to identify the actual use that contractors and DOTs would potentially give to a test like this to take that as a basis in its development early on.

The following questions are here discussed:

- What problem are we trying to solve?
- Why is a field test necessary if there are lab tests available?
- What would a calorimetry test in the field be used for?
- What specific product will be developed?
- Where should the test take place?
- Who could use this product?
- How could the results be interpreted?

What problem are we trying to solve?

It is well recognized that materials, climatic conditions, and construction procedures affect the heat of hydration (HOH) of concrete mixes. The HOH in turn impacts a number of concrete properties including set time, strength, and concrete volume changes that in turn may lead to excessive pavement stresses.

It is believed that there is a need for characterization of concrete mixes in the field in terms of their HOH to provide a way of identifying detrimental situations that could result as a function of changes in climatic conditions, materials, and or construction procedures.

Test procedures are currently available that measure HOH of concrete mixes such as isothermal, adiabatic, and semi-adiabatic equipment. However, the test equipment used is rather expensive and somewhat impractical for application in the field.

Why is a field test necessary if there are lab tests available?

A field test would serve a different purpose than a laboratory test.
A laboratory test may be helpful at the mix design level to characterize mixtures suitable for a specific application. A laboratory test could be also used to catalog mixes for the right application, look for material compatibility, and admixture overdose.

In the field, it is believed that a simple and inexpensive test could be used for process control. This may not necessarily be applicable on a regular basis during paving operations for quality control purposes but could be used every time a change in materials, environment, or construction procedures is expected.

What would a calorimetry test in the field be used for?

A calorimetry test in the field would be used for process control. Several process control scenarios where identified:

1. To identify the right mix for the right conditions (hot/moderate/cold weather). For this scenario,

a test would be needed every time a change in weather is expected (i.e. a change from hot to cold season). This information would help make decisions on suitability of the specified mix for specific weather conditions and adjustments in sawing and curing procedures.

2. Detect compatibility problems. In some instances, it is necessary to make unexpected/unanticipated changes in materials or suppliers (e.g. use of different cement or SCM plants, use of a different admixture manufacturer). For this scenario, a test would be needed every time a change in materials/mix proportions is expected. It is also possible that tests could be performed to address batch-to-batch changes in cement shipments.
3. Perform heat evolution tests continuously for quality control purposes. A test for this purpose may be necessary at least once a day. A fast answer would be necessary to be able to make a decision on whether or not stopping paving operations is required. Due to the likely duration of calorimetry tests (at least 12-24h) this scenario is considered unlikely. Other uncertainties may also make taking a decision difficult. Alternatively, this test might allow for curing measures to be modified.
4. Perform calorimetry tests for record keeping. Similar to the above, this scenario would be used for quality control purposes. However, rather than taking decisions on the spot, the records from these tests would be able to provide some insight after the fact whenever a problem arises.
5. Another application would be to use this test to validate maturity curves in the field.
6. Finding a window for sawing is another potential use.

It is here noted that from a personal communication with Dr. Rupert Springenschmid, president of the RILEM 119-TCE technical committee, it was learned that, in Germany, calorimetry tests (adiabatic temperature rise) are used along with temperature-stress testing (cracking frame) for assessing cracking risk of mass concrete. According to his experience, "neither maximum temperature rise nor maximum temperature of the concrete can give a sound basis for an assessment of cracking risk."

What specific product will be developed?

Three different types of equipment were identified.

1. An isothermal calorimeter.
2. A semi-adiabatic calorimeter.
3. An adiabatic calorimeter.

Advantages and disadvantages for each type of equipment were also discussed. It is believed that the type of test equipment selected will be a function of practicality, accuracy, and cost. These factors will be investigated later on.

Where should the test take place?

Besides the lab test, two locations were identified for the field test: at the point of mix delivery (paving site) and at the plant. Several factors appear to favor locating the test at the plant. These factors include:

- Performing the test at the plant may give some lead-time to take a decision on the delivered batch.
- It may be easier to protect if located at the batch plant or field office, less ruggedability needed.
- It may be easier to isolate from climatic factors.

Who could use the product?

Contractors and/or Field Engineers, field technicians.

How could the results be interpreted?

- There are currently models to characterize HOH curves. A simpler index such as a "heat evolution number "HEN" could be used to characterize mixtures based on HOH curve parameters.

- HOH tests could be performed for input into HIPERPAV. This may be done at the start of the project and whenever a change in materials/climate occurs.
- Whatever the method, it is clear that the HOH test procedure should take into account test variability, materials variability, and other factors that may affect the test results.

Other technical issues discussed at the meeting that will have to be addressed during the development of this test are summarized below:

- Equipment Size
- Sample size
- Weight
- Ruggedness
- Price
- Safety
- Technician level
- Availability by multiple vendors
- Replicates
- Variability
- Identification of factors that will have an effect on the HOH results
- Accuracy/Precision
- Calibration
- Equipment options
- Specification development

APPENDIX C: EXISTING SIMPLE CALORIMETER DEVICES

1. Dewar Devices

Sample size is 2" x 4", or 3" x 6", depending upon the capacity of the Dewar Device.
The steel Dewar Device requires a cork stopper.

Table C1. Dewar device comparison

	ID	OD	D	Ht	Price
Dewar Flask	4.7	5.9	7.5	9.9	173.00
Metal Dewar	3.3	4.6	7.3	9.0	197.00
Metal Dewar	4.2	5.6	8.1	10.5	220.00
Steel Dewar	3.9	4.8	6.2	6.9	129.04
Cork Stopper					14.15

Dewar Flask

Metal Dewar

Steel Dewar

Figure C1. Types of dewar (www.coleparmer.com)

2. Coffee Cup Test Setup

The cement paste is poured into the coffee cups and the temperatures of the samples are recorded for the first ten minutes (price: $2500-$3000).

Figure C2. Coffee cup test setup (Gary Knight, Holcim, 10/1/2004 TWP meeting)

3. Lafarge Basket Test

A 4"x 8" concrete sample was placed in a basket insulated by spray foam, where temperature development was then measured.

Figure C3. Lafarge basket test setup

4. Semi-adiabatic Calorimeter

Sample Size: 6" x 12" and 4" x 8" for concrete; 2" x 4" for paste and mortar

Figure C4. Semi-adiabatic calorimeter–IQ drum (www.quadreliservice.com)

APPENDIX D: EXISTING SENSORS FOR HEAT AND TEMPERATURE MEASUREMENTS

1. Fisher Company (www.fishersci.com)

Temperature data logger: $94.66
Cable and Software: $27.43
Thermocouple temperature range is -200°C to +982°C (-328°F to +1800°F) with ±0.5°C (±0.9°F) accuracy and 0.1 resolution; second channel records ambient temperature from -40°C to +80°C (-40°F to +176°F) with ±0.5°C (±0.9°F) accuracy and 0.1° resolution. Dimensions: 5/8L x 21/4W x 13/8H inches (15 x 56 x 36 mm).

2. QuadreliService and Omega Company (www.omega.com)

OM-CP-OCTRTD
8-Channel Temperature Datalogger: $999
4-Channel Temperature Datalogger: $599
100PT RTD Input; 0.01^{o}C resolution; Real Time Operation
Programmable Start Time; User Calibration through Software
Measurement Range: -200°C to 850°C (-328°F to 1562°F)
Specified Accuracy Range: -200°C to 850°C (-328°F to 1562°F)

OM-CP-QUADTEMP
4 Channel Temperature Datalogger: $ 599
4 Thermocouple Channels and 1 Ambient Channel
Real Time Operation
Automatic Cold Junction Compensation and Linearization
Calibrated Accuracy: +0.5°C (0°C to +50°C)
Temperature Resolution: 0.1°C
Temperature Range: -40°C to 80°C (-40°F to 176°F)

OM-CP-OCTTEMP
8 Channel Thermocouple based Temperature Data Logger: $ 999
Automatic Cold Junction Compensation; Programmable Start Time
8 Thermocouple Channels and 1 Ambient; User Calibration through software
Automatic Thermocouple Linearization;
Temperature Resolution: 0.1°C Temperature Range: -40°C to 80°C
Temperature Accuracy: +0.5°C (0°C to 50°C)

OM-CP-RTDTEMP101 and OM-CP-RTDTEMP110
Precision Temperature Dataloggers : $ 399
User Calibration through
Temperature Measurement Range: -200°C to 850°C (-328°F to 1562°F)
Specified Accuracy Range: -125°C to 600°C (-193°F to 1112°F)
Calibrated Temperature Accuracy: +0.05°C (0.015.) (does not include RTD error)
Temperature Resolution: 0.01°C
Temperature Calibration: digital calibration is available in software

OM-CP-TC4000 and OM-CP-TC110
Ambient Temperature and Thermocouple Loggers: $ 199
Dual Channel Ambient and Remote Thermocouple
User Calibration through Software
Temperature Accuracy: +0.5°C (0 to 50°C)
Temperature Resolution: 0.1°C
Temperature Range: -40 to 80°C (-40°F to 176°F)

OM-CP-TEMP100, OM-CP-TEMP101 and OM-CP-TEMP110
Temperature Dataloggers : $ 109
Memory Size: 32,768 Readings
User Calibration through Software
Temperature Resolution: 0.1°C
Temperature Range: -40°C to 80°C (-40°F to 176°F)