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**ROAD MAP TRACK 1**  
Performance-Based Concrete  
Pavement Mix Design

**PRIMARY SOURCE**  
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Construction Practices for  
Concrete Pavements: A  
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Moving Advancements into Practice (MAP) Briefs describe promising technologies that can be used now to enhance concrete paving practices. MAP Brief 1-4 provides information relevant to Track 1 of the CP Road Map, Performance-Based Concrete Pavement Mix Design.

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## “Moving Advancements into Practice”

### MAP Brief 1-4:

Describing promising technologies that can be used now to enhance concrete paving practices

# Potential Materials Incompatibilities in Concrete Mixtures

## Introduction

Some combinations of concrete materials may be prone to problems with setting and stiffening. Such problems can occur even if all materials meet their individual specifications and perform well when used alone or with other materials. This phenomenon is generally known as incompatibility.

Incompatibility is important because changes in the chemistry or temperature of materials can make a mixture that is acceptable in one batch of concrete behave in an unacceptable way in the next batch, causing problems in placing, compacting, and finishing that are often perceived to be unpredictable and uncontrollable.

Incompatibility is likely occurring because contractors are using increasingly complex combinations of cementitious materials, chemical admixtures, and other materials while asking more of the concrete. The sections are thinner, placing rates are higher, turnaround times are faster, strengths are higher, and the construction season is starting earlier and ending later so that concrete is being placed in more extreme weather conditions. It is also common for materials sources to be changed on a given project without trial mixes or materials tests.

There is no single mechanism behind the wide range of effects that are occurring. Many of the mechanisms are complex and interactive and may require expert evaluation if they occur in the field. Results of incompatibility may include one or more of the following:

- The concrete stiffens much too quickly, preventing proper consolidation or finishing work.
- The concrete sets and gains strength before joints can be cut.

- The concrete does not set in a reasonable time, increasing the risk of plastic shrinkage cracking and late sawing.

For example, a mixture contains Class C fly ash, portland cement, and chemical admixtures, and the combined chemistry of the system causes accelerated stiffening and setting. When one of the materials—fly ash, cement, or admixture—is changed, the concrete setting behavior is normal. Cases have been reported where the mixture is satisfactory at 70°F but cannot be compacted in the paver at 80°F.

Incompatibility issues related to stiffening and setting are generally the result of a sulfate imbalance, although other factors can contribute.

## Sulfate-Related Setting and Stiffening

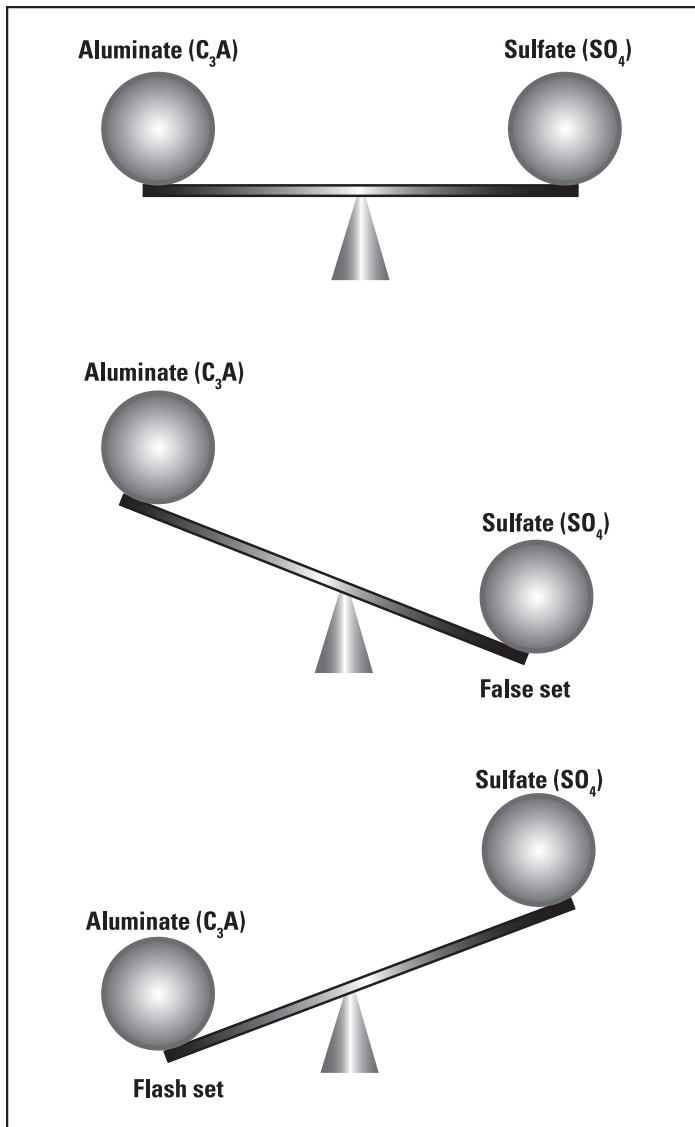
Cement hydration in the first 15 minutes is a delicate balance between the tricalcium aluminate (C<sub>3</sub>A) and sulfate in solution. If the balance is right, the sulfate controls the hydration rate of tricalcium aluminate. If the balance is not right, stiffening and setting problems can occur.

### Too Much or Too Little Sulfate

The amount of sulfate in solution in hydrating cement is critical.

### Flash Set

If there is insufficient sulfate in solution, the tricalcium aluminate reacts quickly with water to form calcium aluminate hydrate (CAH). This reaction generates a large amount of heat, and the fast buildup of calcium aluminate hydrate results in flash set: immediate and permanent hardening of the mix.



Early cement reactions are a balance of tricalcium aluminate (C<sub>3</sub>A) and sulfate (SO<sub>4</sub>) in solution. Excess of either will cause unexpected setting. (Figure courtesy of CTLGroup)

### False Set

Too much sulfate in solution may precipitate out as solid gypsum, causing temporary stiffening of the mix, or false set. If mixing continues, the gypsum will re-dissolve and the stiffening will disappear.

### Form of Sulfate

The form of calcium sulfate (that is, gypsum [CSH<sub>2</sub>], plaster [CSH<sub>1/2</sub>], or anhydrite [CS]) in the cement is also critical, because it affects the amount of sulfate ions in solution. Plaster dissolves faster than gypsum and is therefore useful in preventing flash set; however, cement containing too much plaster will result in too much sulfate in solution, upsetting the balance and resulting in false set, as discussed above. The presence of anhydrite may also upset the delicate balance required because it dissolves slowly and may provide insufficient sulfate in solution to control cements with high tricalcium aluminate (C<sub>3</sub>A) contents.

## Other Factors

Other factors that can affect stiffening and setting incompatibilities include silicate reactions, supplementary cementitious materials, water reducers, cement fineness, temperature, and the water-cementitious materials ratio.

### Silicate Reactions

Most of the strength development in concrete is due to hydration of the silicates in cementitious materials to form calcium silicate hydrate (C-S-H). In general, these reactions start when calcium ions are supersaturated in the solution. If calcium has been consumed during the initial stages of hydration (such as in uncontrolled tricalcium aluminate [C<sub>3</sub>A] hydration), then it is possible that the silicate reactions (and so setting) may be significantly retarded. Silicate reactions can also be affected by the presence of other materials in the mixture, as discussed in the following paragraphs.

### Effects of Supplementary Cementitious Materials

In general, supplementary cementitious materials (SCMs) tend to retard silicate hydration rates, partially due to dilution and partially due to changes in the chemical balances of the system. Hydration is normally extended, however, leading to strength gain beginning more slowly but continuing longer. An SCM containing additional tricalcium aluminate (C<sub>3</sub>A) (typically high-calcium fly ash) can compromise the aluminate-sulfate balance, causing or exacerbating stiffening and setting problems. It may therefore be desirable to use factory blended cements rather than site-blended cements because the manufacturer can optimize the sulfate form and content for the whole cementitious system.

### Effects of Water Reducer

Some water-reducing admixtures will interfere with the hydration rates of cement compounds. Lignin-, sugar-, and triethanolamine (TEA)-based products (normally Type A or B admixtures based on ASTM C 494 classification) have the combined effect of accelerating aluminate reactions and retarding silicate reactions. The use of such an admixture may tip a marginally balanced cementitious system into incompatibility. Some (Type A) water reducers accelerate aluminate reactions. A system that has just enough sulfate to control normal aluminate reactions, can therefore be thrown out of balance. Aluminate reactions are then uncontrolled and workability is reduced. Adding more admixture with the mixing water to boost workability likely exacerbates the problem, possibly leading to an overdose with its attendant problems (such as retardation of the silicate reactions).

One solution is to delay adding the water-reducing admixture until the early aluminate reactions are under control.

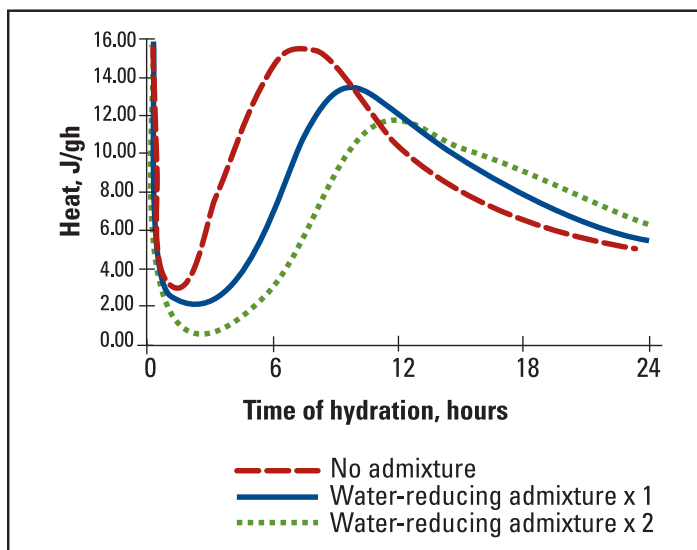
The length of the delay will have to be determined for the specific mixture. In addition, the same water-reducing admixtures retard the silicate reactions, delaying setting and slowing strength gain. It is therefore feasible that a system containing certain water reducers will exhibit classic early stiffening because of uncontrolled aluminate hydration, followed by severe retardation of final set because of slowed silicate reactions. This has been observed in the laboratory and in the field.

**Effects of Cement Fineness**

Cement fineness influences reaction rates. The finer the cement, the greater the rates of reaction and the greater the risk of an unbalanced system. Finer cements require a higher sulfate content and perhaps a higher plaster-to-gypsum ratio.

**Effects of Temperature**

The solubility and reactivity of all of the compounds are strongly influenced by temperature, with higher temperatures generally increasing solubility (except calcium) and accelerat-



**Plot of heat generated by cement hydration of cement pastes containing varying amounts of lignosulfonate-based water-reducing admixture (Figure courtesy of CTLGroup)**

ing reaction rates. Increasing temperature, on the other hand, decreases the solubility of calcium sulfate, thus reducing the amount of sulfate in solution available to control the accelerated aluminate reactions and thereby potentially making the system unbalanced. A change of as little as 10° can tip a mixture from being workable to exhibiting early stiffening. In warmer weather, more sulfate (plaster) is needed to control rapid tricalcium aluminate (C3A) reactions.

**Effects of Water-Cementitious Materials Ratio**

The severity of these effects is also related to the ratio of water to cementitious materials. Lower water contents effectively mean that the cement grains are closer together; therefore, the early hydration products have to fill less space before stiffening results, so early stiffening may occur. The same mixture at a higher water-cementitious materials ratio, with greater particle spacing, may not exhibit the same problems.

**Testing for Potential Incompatibilities**

The most reliable way to detect whether a mixture is likely to be problematic is to conduct a series of tests and trial batches on the materials at the field temperature. Laboratory tests must be run sometime before construction begins to prequalify materials planned for the project. Field tests (materials acceptance tests) should be run as materials are delivered to the site to ensure that site materials are similar to prequalified materials. Preferably, materials-acceptance tests should be run the day before batching is planned, although at the height of construction, results may be needed within a few hours.

**What to Test**

A suggested test protocol is summarized in the table below.

In many cases, there is no single pass/fail limit because what is acceptable in one system or environment is not acceptable in another. It is recommended that test results be tracked over time, and a significant change in a test result will indicate potential problems. It is recommended that as many of these tests as practical be conducted at the prequalification

**Recommended Tests and Their Applications (Taylor 2006)**

	<b>Stiffening and setting</b>	<b>Cracking</b>	<b>Air-void system</b>
Laboratory tests	<ul style="list-style-type: none"> <li>• Materials chemistry</li> <li>• Calorimetry</li> <li>• Minislump</li> <li>• Rheometer</li> <li>• Time of set (ASTM C 191)</li> <li>• Stiffening (ASTM C 359)</li> </ul>	<ul style="list-style-type: none"> <li>• Materials chemistry</li> <li>• Ring test</li> <li>• Time of set</li> </ul>	<ul style="list-style-type: none"> <li>• Materials chemistry</li> <li>• Air-void analyzer</li> <li>• Hardened air (ASTM C 457)</li> <li>• Clustering index</li> </ul>
Field tests	<ul style="list-style-type: none"> <li>• Slump loss</li> <li>• Time of set (ASTM C 403)</li> <li>• Ultrasonic P-wave</li> <li>• Semi-adiabatic temperature measurement</li> </ul>	<ul style="list-style-type: none"> <li>• Time of set</li> <li>• Semi-adiabatic temperature measurement</li> <li>• Ultrasonic P-wave</li> </ul>	<ul style="list-style-type: none"> <li>• Foam index</li> <li>• Foam drainage</li> <li>• Unit weight (ASTM C 138)</li> <li>• Air content (ASTM C 231)</li> <li>• Air-void analyzer</li> </ul>

stage so that a point of reference is available for comparison with tests conducted during construction. A test result that is out of the ordinary may be a problem with the testing or with the material. Such data should therefore be reviewed with an understanding of limitations and potential errors in testing. Interpreting the results may require expert input.

The decision about how many of these tests to conduct will be based on the economics of the project, including the value of the project, probability of failure, cost of testing, and cost of failure. Many problems will be avoided by regularly monitoring slump loss, unit weight, set time, and admixture dosages. Significant changes in any of these parameters will indicate the need for more intensive examination of the materials at hand.

### Central Laboratory (Prequalification) Tests

Factors to monitor generally include the following.

**Minislump.** The minislump cone test monitors the area of small slump cone samples made using paste at selected time intervals. The test is effective at identifying systems prone to early hydration problems (Kantro 1981). Reproducibility between labs is reportedly low.

**Isothermal Calorimetry.** The energy required to maintain a hydrating paste mixture is monitored in an isothermal calorimeter. Changes in the timing or magnitude of the temperature rise, or the shape of the heat-energy-versus-time plot, will flag potential problems in the silicate reactions (Wadso 2004). ASTM 1679 describes a method for this test.

**Shear Stress Increase.** Measurement of the shear stress increase with time in a parallel plate rheometer using paste is showing promise as a method to monitor silicate hydration processes.

**Rate of Stiffening.** The ultrasonic P-wave test allows measurement of the rate of stiffening of a mixture in the lab and the field.

**Early Stiffening.** The method described in ASTM C359 / AASHTO T185 is being used in some laboratories to indicate potential problems in the early aluminate reactions. Interpretation of the results must be undertaken with care and understanding. ASTM is developing a practice for this application.

### Field Laboratory (Monitoring) Tests

Factors to monitor generally include the manufacturer's mill test x-ray data, semi-adiabatic temperature, concrete slump loss and setting time, uniformity of the air-void system, and air-void clustering.

**X-Ray Data from Manufacturer's Mill Test Report or Mill Certificate (ASTM C114, ASTM C1365).** This test monitors the chemistry of the cement and fly ash. Changes in calcium, tricalcium aluminate (C3A), sulfate (SO<sub>3</sub>), alite (C3S) or belite (C2S) may indicate problems.

**Semi-Adiabatic Temperature Measurement.** This test monitors the temperature of paste, mortar, or concrete mixtures in sealed containers (Dewar flasks, insulated cups, or proprietary equipment). Changes in the timing or magnitude of the temperature rise, or the shape of the heat-versus-time plot, will flag potential problems in the silicate reactions. ASTM is working on developing a standard method for this test.

**Concrete.** For this test, make concrete batches and monitor slump loss with time as well as setting time (ASTM C143).

## Potential Solutions to Incompatibilities

If problems are observed in the tests or in the field, then one or more of the following actions may resolve them:

- Reduce the concrete temperature by cooling the materials and/or working at night.
- Seek a fly ash with lower calcium content.
- Reduce fly ash dosage.
- Delay admixture addition.
- Change the type of chemical admixture.
- Change the source of cement.
- Increase mixing time.
- Seek expert advice to establish what the root cause of the problem is so that the correct remedial action can be taken.

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