Concrete Mixtures and Optimum Performance: Avoiding “Incompatibility” of Components

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“Incompatibility” as per our experiences

- Abnormal **setting** and/or **strength gain**
  - Either or both, usually both
  - May also involve unusual slump loss
  - Trends often “counter-intuitive”

- Sulfate related phenomena
  - Abnormal early chemistry interactions are evident using calorimetry of fresh mortar or paste
  - Early strengths confirm
  - Literature review - similar sulfate imbalance issues reported by others
“Incompatibility” as per our experiences

• Specific abnormal behavior:
  – Extreme set delays, some over 24h, or…
  – In extreme cases (less common), flash set (!)
  – Unusual slump loss sometimes reported
  – Very poor early strengths, in all cases
• Has often gone undiagnosed
• May suddenly occur after a source change
• Can come and go mid-project with “in-spec” materials and no source change
Observed influences

- High temperatures
- Class C fly ash content
- Chemical admixture selection
- Chemical admixture dosage
- Cement sulfates – amount and form
- Normal variability of materials
Related cement chemistry

- Sulfates ($\text{SO}_3$) from gypsum control set time by reacting with $\text{C}_3\text{A}$ to form insulating byproducts.
- Uncontrolled early $\text{C}_3\text{A}$ hydration results in:
  - Unpredictable set effects and/or slump loss
  - Interrupted silicate hydration & strength gain
- Cement is usually the only source of soluble sulfates in concrete.
- Class C fly ash, admixtures, and higher temps increase demand for readily soluble sulfates in concrete without increasing available sulfates.
Case history of an “incompatibility” issue

- Early summer, 2003
- Mississippi concrete producer with multiple plants
- Common mix characteristics:
  - Class C fly ash @ 20% or 25%
  - Type A/B/D water reducer @ 4 to 6 oz/cwt, all mixes
  - Concrete temps: 90º F

Concrete field issues reported by the ready-mix producer:
- 1-day strengths on bridge deck pours suddenly fell by 60-70%
- Set times extended dramatically, to almost 48 hours in some cases
- Very low strengths through 7 days reported on most mixes
- Excessive cracking issues on some jobs
- Duration of issue less than 10 days, performance returned to normal
- No materials sources were changed at any plants
Mortar cube testing for influence of cement

- Cements from 11 plants around the US
- 25% class C fly ash
- Type A/B/D water reducer @ 6 oz/cwt dose
- 90° F material & initial cure temps
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Calorimetry: a simple diagnostic test method

Thermal measurements of fresh paste or mortar can be used to document early hydration effects and to predict abnormal behavior of a concrete mix design.

- Simple, inexpensive thermocouple & data logger systems for “semi-adiabatic” calorimetry can produce results similar to isothermal systems.
Testing program for incompatibility influences

Mixtures with class C ash simulating field concrete with admixtures & dosages as used for retardation

Heated materials - representative summer temps
Modified C 109 mortar cubes and semi-adiabatic calorimetry

Materials & test variables

Cements:
- Type II “reference” cement produced @ different SO₃ levels via varying gyp feed to the mill
- 2 “problem” cements (under-sulfated)

Class C fly ash:
- 25% replacement (default)
- Some mixes @ 10% or 35%

Mixture temps: 73°, 90°, 100° F

Admixtures & default dosages:

<table>
<thead>
<tr>
<th>Admixture</th>
<th>Description</th>
<th>Dosage</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSwr</td>
<td>Corn syrup-based water reducer</td>
<td>6 oz/cwt</td>
</tr>
<tr>
<td>LwrA</td>
<td>Lignin-based water reducer “A”</td>
<td>6 oz/cwt</td>
</tr>
<tr>
<td>LwrB</td>
<td>Lignin-based water reducer “B”</td>
<td>6 oz/cwt</td>
</tr>
<tr>
<td>LwrC</td>
<td>Lignin-based water reducer “C”</td>
<td>8 oz/cwt</td>
</tr>
<tr>
<td>CSretA</td>
<td>Corn syrup-based retarder “A”</td>
<td>3 oz/cwt</td>
</tr>
<tr>
<td>CSretB</td>
<td>Corn syrup-based retarder “B”</td>
<td>3 oz/cwt*</td>
</tr>
</tbody>
</table>
Test procedures

Cube (mortar) batches
- Admix added with water
- Water adjusted for constant flow (105 to 115)
- Standard sand
- All materials pre-heated to specified temp
- Cubes returned to heat for 18-24 hours, then standard cure

Calorimetry (paste) batches
- Admix added with water
- w/cm = 0.40, all mixtures
- 2 minutes mixing time
- Materials pre-heated, samples returned to heat environment during data collection
- Thermocouple inserted within 60 sec of completion of mixing
Controlled environments for pre-heating materials and maintaining cure temps.
Examples of “normal” hydration & strength development, mixtures with 25% fly ash, with or without various admixtures, 73° or 90° F initial temps

**Graph:**
- **Y-axis:** Compressive strength, psi
- **X-axis:** Hydration time, hours
- **Legend:**
  - 1 day
  - 3 days
  - 7 days
  - 28 days

**Graph:**
- **Y-axis:** ΔT (Sample - Tambient), °F
- **X-axis:** Hydration time, hours
- **Legend:**
  - 3.3-roadmix
  - 3.7-CSwr
  - 4.1-CSwr
  - 3.3-LwrA
  - 3.3-CSretA
Data comparisons – sensitivity of variables
(cubes and calorimetry)

1) Effects of temperature and sulfate level
2) Effects of Class C fly ash content
3) Comparisons of different admixtures in “moderate” conditions
4) Comparisons of different admixtures and dosages in more “extreme” conditions
5) Example mixtures with “problem” cements
Effects of initial temperature and cement SO₃ level, mixtures with CSwr @ 6 oz/cwt, 25% fly ash, initial temps 73°, 90°, or 100° F

No corresponding calorimetry for 73° cube batches.

ΔT (Tsample – Tambient), °F

Hydration time, hours

compressive strength, psi
Effects of fly ash content, mixtures with CSwr @ 6 oz/cwt, 90º F initial temp, SO₃ = 3.3 or 3.7%

![Graph showing compressive strength over hydration time for different fly ash contents and SO₃ levels.](image)
Admixtures compared in similar mixtures, “moderate” conditions, with 25% fly ash, 90° F initial temp, SO3 = 3.3%

[Graph showing compressive strength and hydration time for different admixtures.]
Admixture comparisons & dosage effects – various “extreme” condition mixtures with 25% fly ash, initial temps 90 or 100° F, sample ID: admix - dose/cwt - SO₃ - temp (° F)

- ΔT (Tsample – Tambient), °F
- Compressive strength, psi
- Hydration time, hours
Admixture comparisons & dosage effects – various “extreme” condition mixtures with 25% fly ash, initial temps 90 or 100º F, sample ID: admix - dose/cwt - SO₃ - temp (º F)

Expanded first hour of hydration

Flash set
“Problem” cements (imports) in various mixtures with fly ash and CSwr, sample ID: cement(SO₃) - ash% - admix dose/cwt - temp (°F)
“Problem” cements (imports) in various mixtures with fly ash and CSwr, sample ID: cement(SO₃)-ash%-admix dose/cwt-temp(°F)

- Compressive strength, psi
- Hydration time, hours

Expanded first hour of hydration

Flash set

Graph showing hydration time and temperature differences.
Conclusions & recommendations

- Key influences – at least 5 factors
- Problem resolution often = change any one
- Extreme cases may need several changes
- Most immediate relief: fly ash %, admixture dose
- Higher cement SO$_3$ or more soluble form will be helpful but may not be possible within spec limits
- Changing admixture type may help or resolve
- High doses of A/B/D water reducers for retardation should be avoided
- Simple test methods can be used to investigate
Questions?

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