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Spotting Incompatibility Problems in Concrete Mixtures

Session # 52

 

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Courtesy of PCA

Background

- Durability concerns and increasing performance requirements often lead to higher levels of complexity when selecting proportions and types of materials for use in concrete mixtures.
- Increased number of materials combinations may lead to undesirable interactions (incompatibilities) between various ingredients of the mixture (concrete becomes less forgiving).

Types of Admixtures

- ASTM C260- Air Entraining Admixtures (AEA)
- ASTM C494 – Chemical admixtures
  - *Type A* – Water-reducing (min 5% water reduction)
  - *Type B* – Retarding
  - *Type C* – Accelerating
  - *Type D* – Water-reducing and retarding (min. 5%)
  - *Type E* – Water-reducing and accelerating (min. 5%)
  - *Type F* – Water-reducing, high range (min.12% water reduction); **also called HRWR or SP**
  - *Type G* – Water-reducing, high range and retarding (min.12%)
  - *Type S* – Specific performance (i.e. VMA, corr. inhibitors)

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Typical components of cement

Alite (~C_3S) - tricalcium silicate $3CaO \cdot SiO_2$	**~60%**
Belite (~C_2S) - dicalcium silicate $3CaO \cdot SiO_2$	**~15 %**
Tricalcium aluminate (~C_3A) $3CaO \cdot Al_2O_3$	**~10 %**
Tetracalcium aluminoferrite (~C_4AF) $4CaO \cdot (Al_2O_3)_X \cdot (Fe_2O_3)_Y$	**~10 %**
Other phases gypsum- $CaSO_4 \cdot 2H_2O$, hemihydrate - $CaSO_4 \cdot 1/2H_2O$, or anhydrite - $CaSO_4$.	**~5 %**

The Mill Report or Mill Certificate

BUZZI UNICEM USA

PO Box 482-Greencastle, IN 46135-(765) 653-9766

This is to certify that **Type I** meets ASTM C-150 Specifications for Portland Cement.

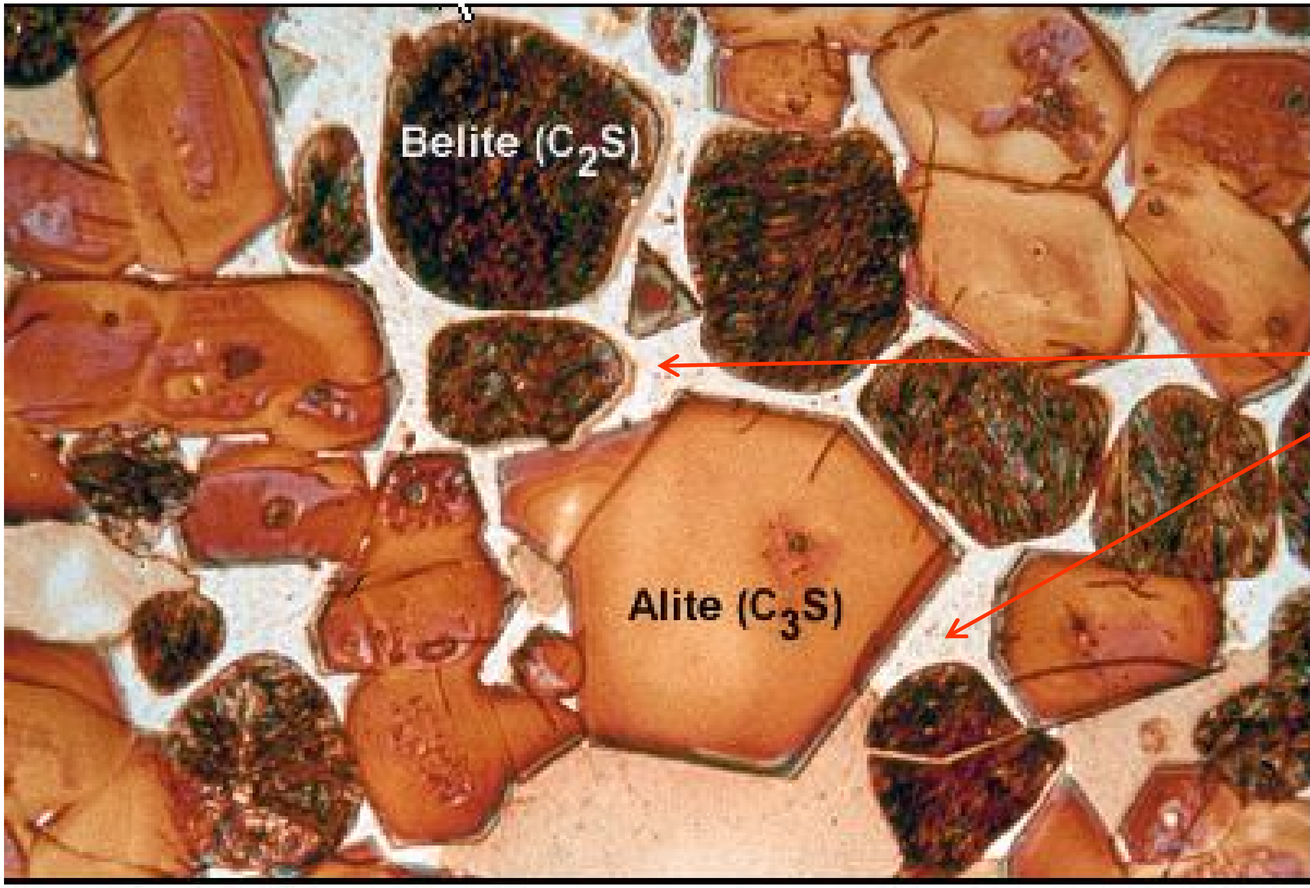
Chemical Data		Physical Data	
ASTM C114		ASTM C185	
Silicon Dioxide (SiO_2)	20.61	Air Entrained (%)	8.2
Aluminum Oxide (Al_2O_3)	5.24	ASTM C204	
Ferric Oxide (Fe_2O_3)	2.33	Fineness (cm^2/gm)	3670
Calcium Oxide (CaO)	65.27	ASTM C151	
Magnesium Oxide (MgO)	1.19	Autoclave Expansion (%)	-0.001
Sulfur Trioxide (SO_3)	3.32	Compressive Strength, PSI	
Loss on Ignition	1.75	ASTM C109 Mortar Cubes	
Sodium Oxide	0.12	1-Day	2370
Potassium Oxide	0.74	3-Day	3970
Insoluble Residue	0.50	7-Day	5020
Total Alkali as Na_2O	0.61	28-Day	
POTENTIAL COMPOUND COMPOSITION		ASTM C191	
		Setting Time:	
Tricalcium Silicate (C_3S)	61	Vicat	
Dicalcium Silicate (C_2S)	13	Initial, Min.	102
Tricalcium Aluminate (C_3A)	10	Final, Min.	221
Tricalcium Aluminoferrite(C_4AF)	7		

Silo	Bill of Lading	Tons	Date	Silo	Bill of Lading	Tons	Date

Typical components of cement

Chemical Data	
ASTM C114	
Silicon Dioxide (SiO_2)	20.61
Aluminum Oxide (Al_2O_3)	5.24
Ferric Oxide (Fe_2O_3)	2.33
Calcium Oxide (CaO)	65.27
Magnesium Oxide (MgO)	1.19
Sulfur Trioxide (SO_3)	3.32
Loss on Ignition	1.75
Sodium Oxide	0.12
Potassium Oxide	0.74
Insoluble Residue	0.50
Total Alkali as Na_2O	0.61
POTENTIAL COMPOUND COMPOSITION	
Tricalcium Silicate (C_3S)	61
Dicalcium Silicate (C_2S)	13
Tricalcium Aluminate (C_3A)	10
Tricalcium Aluminoferrite(C_4AF)	7

Clinker as viewed under the petrographic microscope



Interstitial – mixture of C_3A and C_4AF

Width of field = 0.31 mm

Chemical reactions of cement with water

$$2C_3S + 11H \rightarrow C_3S_2H_8 + 3CH$$

$$2C_3S + 9H \rightarrow C_3S_2H_8 + CH$$

C-S-H gel

Calcium hydroxide

ettringite

$$C_3A + 26H \rightarrow 3C\bar{S}H_2 + C_6A\bar{S}_3H_{32}$$

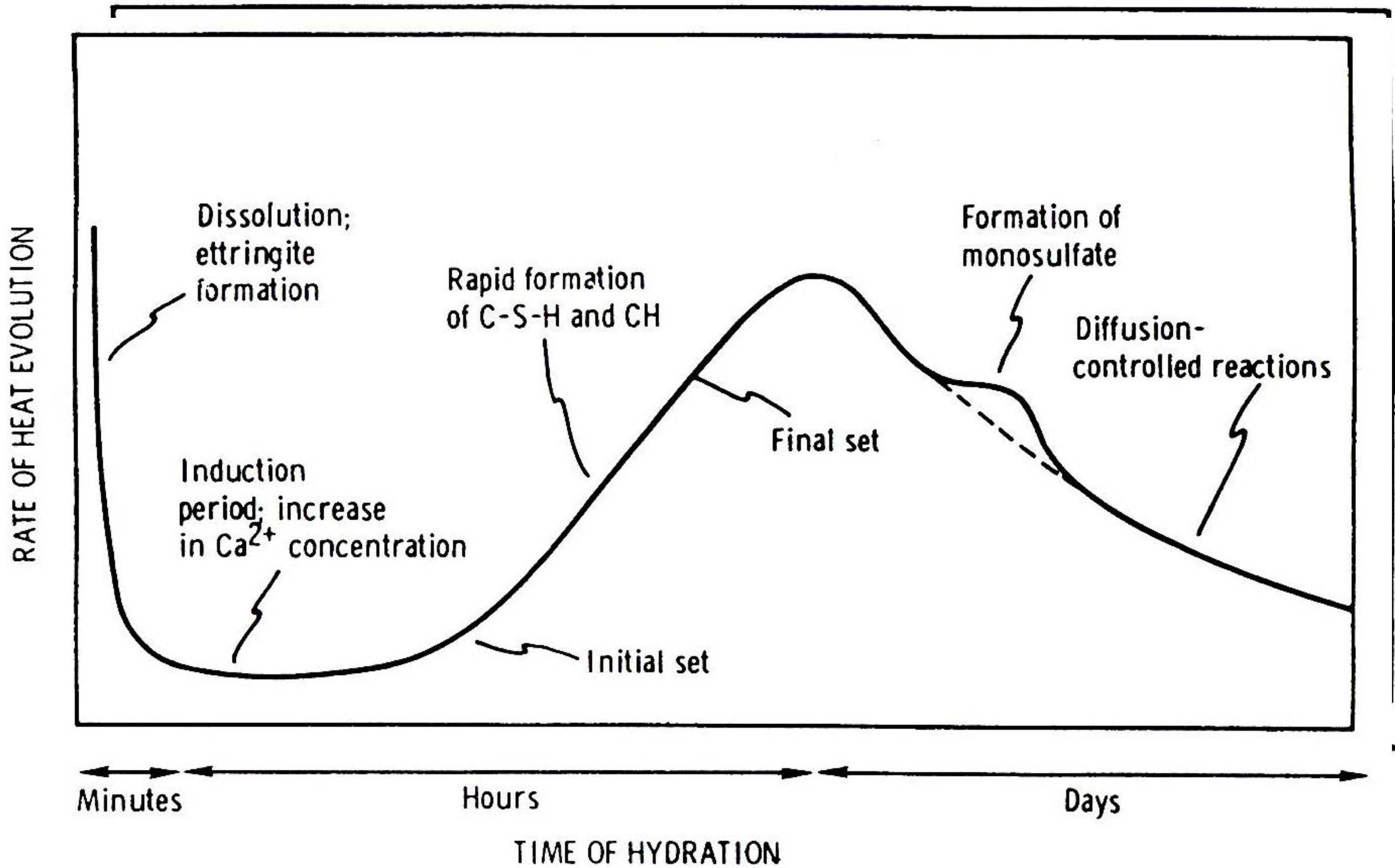
monosulfate

$$2C_3A + 4H \rightarrow C_6A\bar{S}_3H_{32} + 3C_4A\bar{S}H_{12}$$

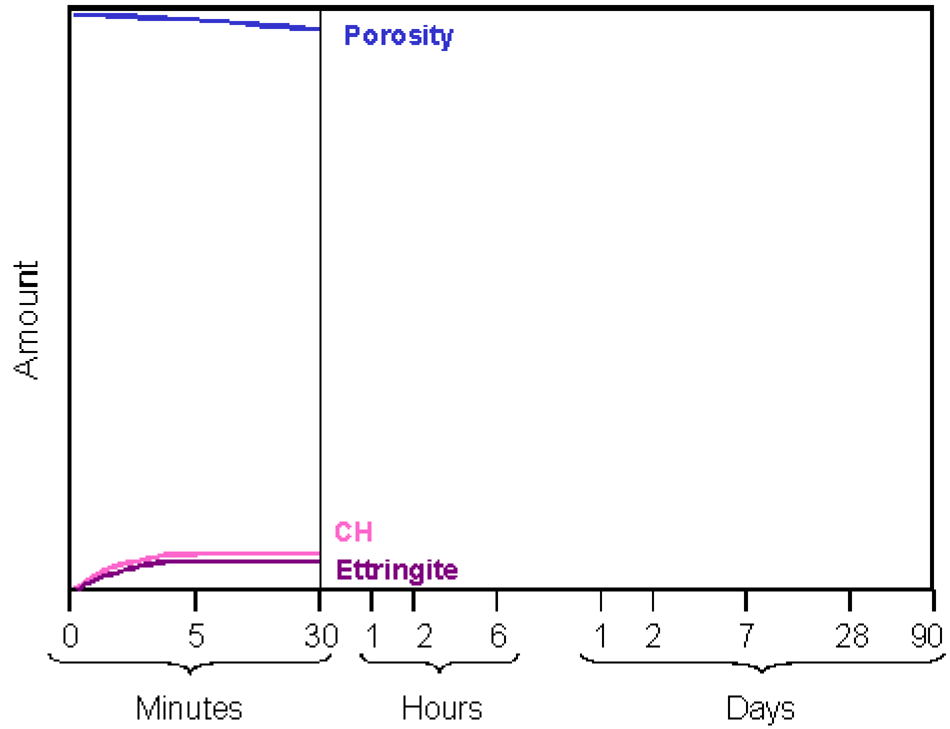
$$3C_3A + 12H \rightarrow CH + C_4AH_{13}$$

$$C_4AF + 10H \rightarrow 2CH + C_6AFH_{12}$$

Rate of Heat Evolution During Hydration of Portland Cement

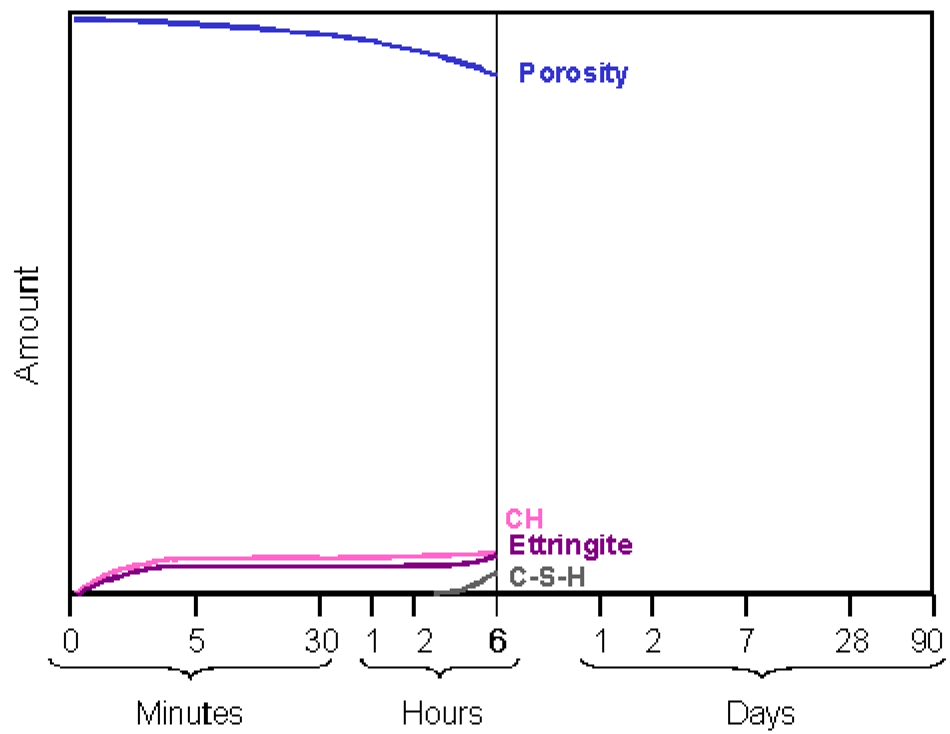


Development of hydration products – kinetics of hydration

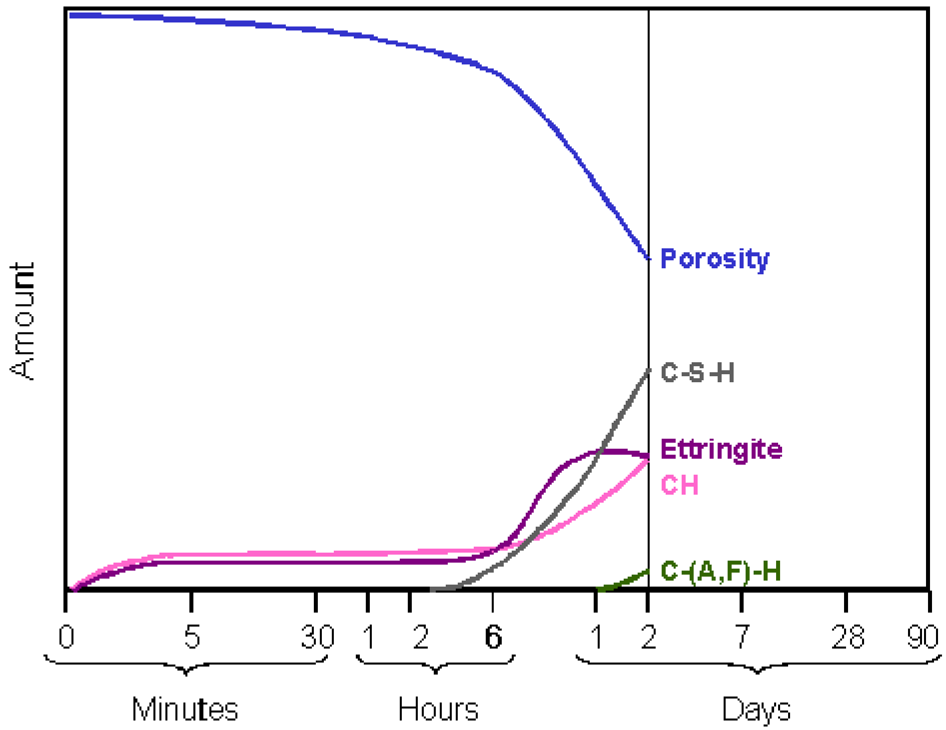


(Locher at al, 1976)

Development of hydration products – kinetics of hydration

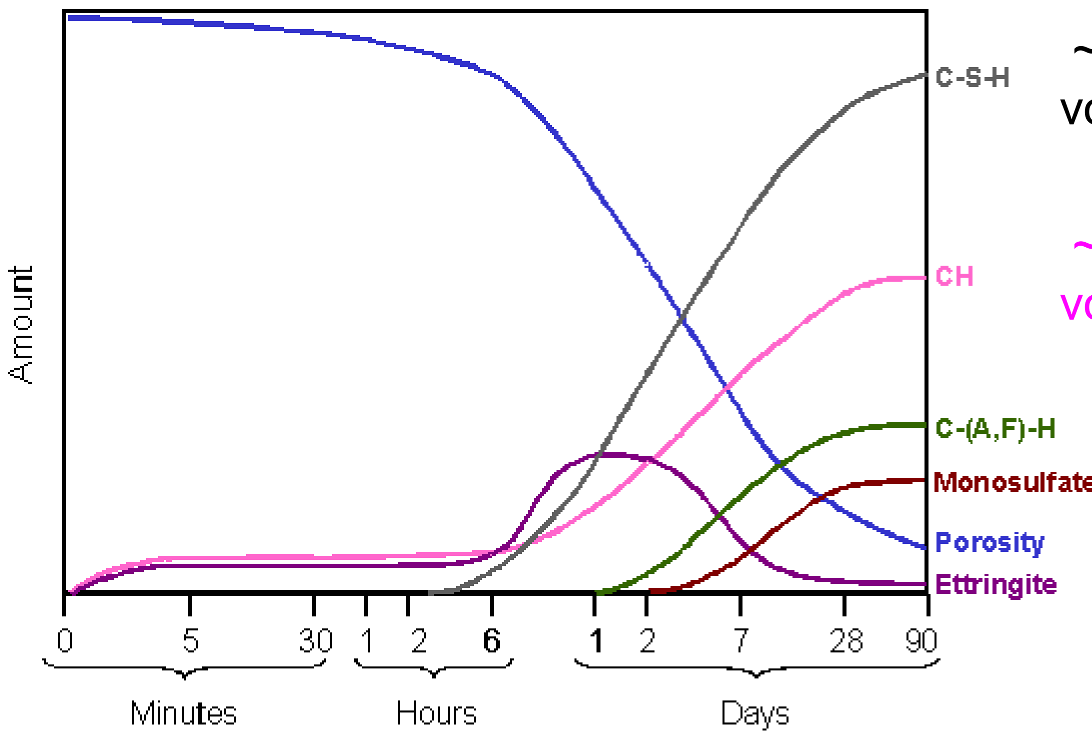


Development of hydration products – kinetics of hydration



(Locher at al, 1976)

Development of hydration products – kinetics of hydration



(Locher at al, 1976)

~50% by volume

~20% by volume

Incompatibility

- The term "incompatibility" has been applied to a variety of different types of abnormal performance of concrete in both plastic and hardened concrete, including
  - Setting and strength gain issues,
  - Increased water demand (less than expected water reduction)
  - Problems with the air void system, and
  - Early age cracking

Incompatibility …

- Other problems, which are expected, will not be considered as incompatibility. For example,
  - cement & silica fume (SF) system
    - higher water demand because of high fineness of SF
    - expected phenomenon ➔ not an incompatibility problem

Incompatibility….

- It is possible for a combination of common materials, each of which individually meets applicable specifications and has been used without problems in other mixtures or under slightly different conditions, to result in completely unexpected and abnormal behavior.
- The mixture in question may represent commonly used proportions, yet result in severely abnormal slump loss or setting tendencies (usually extended delays but occasionally flash set) followed by extremely poor early strength development

V.T Cost and G. Knight, ACI –SP-241.2007, pp. 39-58

Incompatibility….

- Late age strengths of such mixtures may be relatively unaffected though in more extreme cases even 28-day strengths can be significantly lower than expected.
- Such behavior has sometimes occurred in a concrete mixture that performed normally on a project for a period of time and then suddenly experienced these issues though no material source was changed

V.T Cost and G. Knight, ACI –SP-241.2007, pp. 39-58

Incompatibility….

- This suggests that the threshold of incompatibility can be suddenly crossed due to slight, routine variability of one or more materials, methods, or other influences.
- In order to determine the most practical and economical course of action for preventing or dealing with these abnormal behaviors, however, the concrete producer need to
  - Have better understanding of all of the significant (controllable) contributing factors
  - Realize the relative importance of each of these factors

V.T Cost and G. Knight, ACI –SP-241.2007, pp. 39-58

Objectives of the Study

- To study the potential incompatibility problems arising in the cementitious systems containing fly ash and chemical admixtures.

- Incompatibility manifested as abnormal early age stiffening, setting behavior or improper air void system.

Outline

- Objectives
- **Literature Review**
- Details of Experiment
- Results
- Future Work
- Conclusions

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Indiana Experience

- Class C fly ash /non-chloride accelerators combinations when used in mixes placed in northern Indiana resulted in severe set retardation

- High variability in the amount of entrained air and its sensitivity to temperature was observed in the presence of polycarboxylate superplasticizers (HRWR)

Indiana Experience

- Very high dosage of a specific Type F (high range water reducing) admixtures HRWR created excessive retardation of the mix during use in cool weather (i.e. it took weeks to gain strength for construction operations to continue)

- Point of introducing of a specific Type A (water reducing) admixtures during batching sequence was critical for achieving water reduction to obtain the same workability (i.e. slump)

Origin of Incompatibility

Major reasons of incompatibility problems:

- Binder driven incompatibility
  - Cement related incompatibilities - Cement chemistry
  - Supplementary Cementitious Materials (SCMs) related incompatibilities
  - Combination of cement and SCM

Origin of Incompatibility ...

- Admixture related incompatibilities
  - Binder- admixture interactions
  - Admixture – admixture interactions

- Miscellaneous reasons – temperature, delayed addition and/or excessive dosages of admixtures

Cement Driven Incompatibility

- Role of C_3A:
  - Uncontrolled C_3A hydration is the major reason for early stiffening behavior – flash set.
  - Delays setting time by preventing hydration of silicates

Cement Driven Incompatibility …

- Role of sulfates:
  - Sulfates control the early rapid hydration of C_3A
  - Sulfate-aluminate balance is critical for [Sandberg & Roberts, 2005):
    - Performance of admixtures
    - Appropriate workability and strength

Role of sulfates…

- In sulfate deficient systems, the rapid hydration of C_3A yields calcium aluminate hydrates and leads to flash setting
- In the presence of excessive sulfate, nucleation and growth of gypsum crystals can lead to false setting behavior – short mixing time.
- The level of sulfates in the pore solution strongly influences the adsorption of certain chemical admixtures [Prince et al.,2002]

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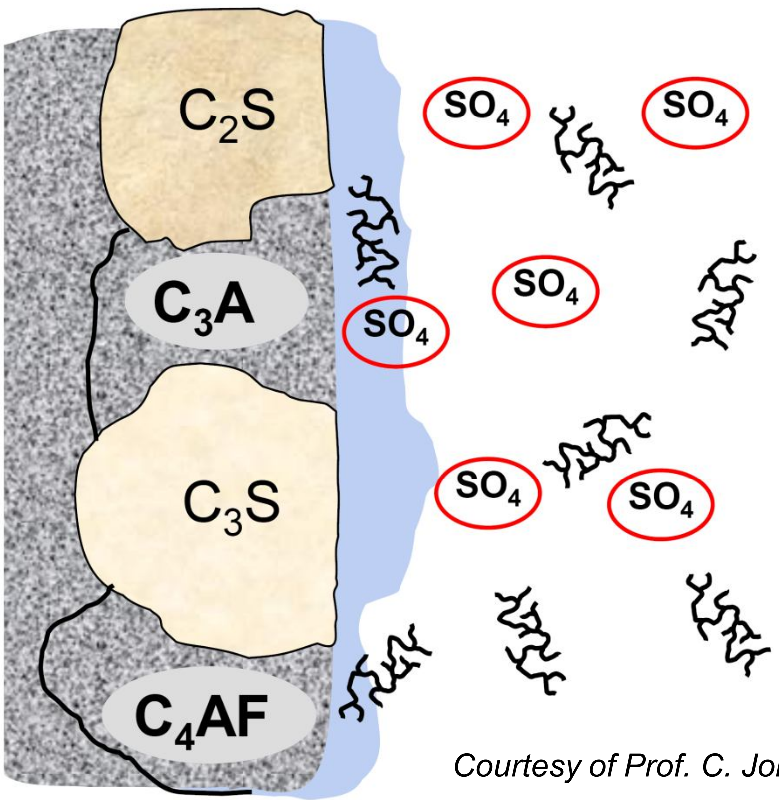
SO_4 Supply / Demand Equilibrium

SO_4 / C_3A Ratio (in first min-hrs)

SO_4/C_3A	Too low	Balanced	Too high
Product formed	CAH gel	Ettringite	Secondary gypsum
Behaviour of concrete	Flash set rapid, irreversible loss of slump	Controlled C_3A hydration, adequate slump retention	False set rapid, reversible slump loss

Courtesy of Prof. C. Jolicoeur, Université de Sherbrooke,Canada

Cement – Admixture Sulfate-related Incompatibility



Competitive adsorption of PNS and SO_4 at most reactive sites of cement particles

Courtesy of Prof. C. Jolicoeur, Université de Sherbrooke,Canada

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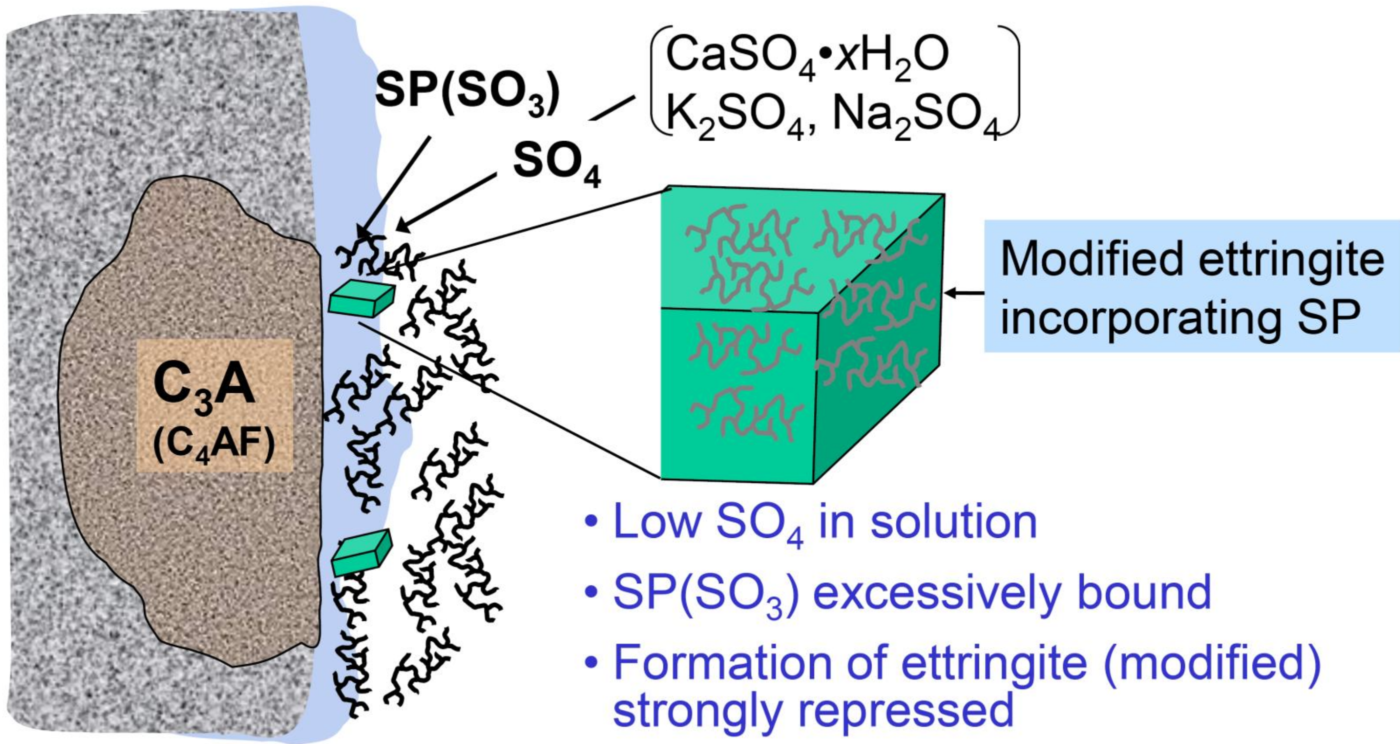
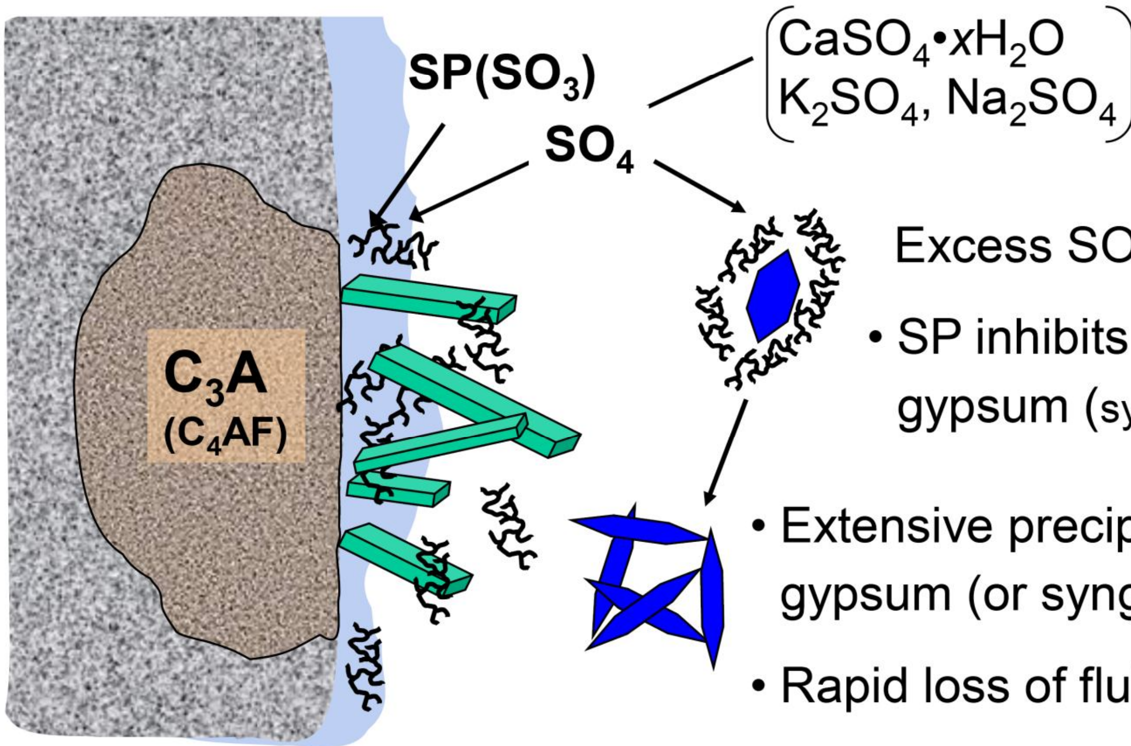
Illustration of Cement-SP Incompatibility
Due to Sulfate Deficiency



- Low SO_4 in solution
- SP(SO_3) excessively bound
- Formation of ettringite (modified) strongly repressed
- Rapid loss of fluidity

Courtesy of Prof. C. Jolicoeur, Université de Sherbrooke,Canada

Illustration of Cement-SP Incompatibility Due to Excess Sulfate

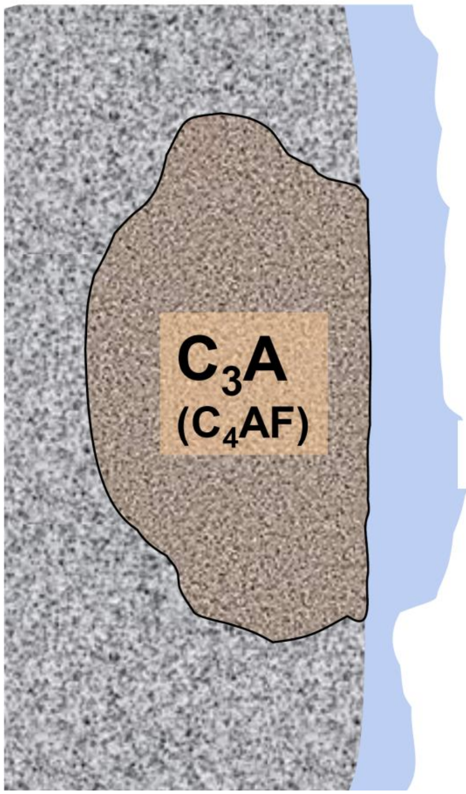


Excess SO_4 in sol^n

- SP inhibits growth of gypsum (syngenite)
- Extensive precipitation of gypsum (or syngenite)
- Rapid loss of fluidity

Courtesy of Prof. C. Jolicoeur, Université de Sherbrooke,Canada

Origin of Cement-SP Incompatibility



SP adsorption

- C_3A content
- C_3A reactivity (type)
- Cement fineness
- SP molecular properties

SO_4 balance in solution

- Type of $CaSO_4$ (G, H, A)
- Alkali sulfate content
- Ettringite formation (rate, type)
- Gypsum (syngenite) precipitation

Courtesy of Prof. C. Jolicoeur, Université de Sherbrooke,Canada

Role of Alkalis in Cement:

- Higher alkali cements react faster ➔ higher rate of stiffening -> higher slump loss and difficulties with air entraining and air stability.
- Jennings et al., [1997] correlated incidents of premature deterioration with alkali contents of the cementitious materials.

Role of Alkalis in Cement…

- Dosage requirement of admixtures are directly related to alkali content of the system.
- Low alkali content systems exhibited lower stability of air void system.
- Low alkali cement & synthetic air entrainer combination resulted in severe strength loss [Cross et al., 2000]

Role of Supplementary Cementitious Materials (SCMs)

Incompatibility problems:

- Jennings et al, [1997] cited the use of Class C fly ash as one of the factors strongly associated with pavement deterioration.
- Concrete with cement + fly ash resulted in excessive slump loss in field concrete [Taylor et al., 2006]
- Class C fly ash prolonged the onset of acceleration phase and hence caused retardation with I and II type cements. [Wang .H et al., 2006]

Role of SCMs …

Role of Fly ash Composition on workability:

- May introduce reactive aluminate phases like C_3A and Klein's compound (calcium sulfo-aluminate)
- Sulfates and alkalis that come along with fly ash may disturb the sulfate balance of the system [Gress, 1997]

Role of SCMs …

- Gebler and Klieger [1983] observed 59% reduction in air content (after 90 minutes) in concretes made with class F ash
- Mixes with class F fly ash required two to five times higher dosage of air entraining agents (AEA) [Zhang,1996]

Role of SCMs …

- Higher AEA requirement is attributed to: [Indrek et al.,2004; Gao et al., 1997]
  - Amount of un burnt carbon;
  - Specific surface area;
  - Accessibility of the surface area;
  - Chemical nature of the surface
- Calcium and magnesium ions in the fly ash affect the air entrainment by forming precipitates with the surfactant AEAs

Admixtures Driven Incompatibility

Admixture – Binder interactions:

- High dosage of AEA with low alkali cement - air void clustering - loss of strength [Taylor et al., 2006]
- Low alkali cement + VR based significant increase in spacing factor (10min vs 90 min) [Cross et al.,2000 , Ansari et al.,2002]

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Admixtures Driven Incompatibility …

Admixture – Binder interactions:

- High Alkali contents in the mix were found to increase the amount of polycarboxylate type of super plasticizers required to obtain optimum fluidity[Hanehara & Yamado, 1999]

- Lignin based admixtures reduce the solubility of sulfates disturbing the C_3A sulfate balance. [Shiping Jian et al.,1999]

Admixtures Driven Incompatibility …

Admixture – Admixture interactions:

- Sugar content of the lignin-based, water-reducers linked to set retardation and air entrainment difficulties [Aitcin et al., 1994]

- VR + Lignin based WRA - high entrapped air & reduction in specific surface area [Bedard and Mailvaganam, 2006]

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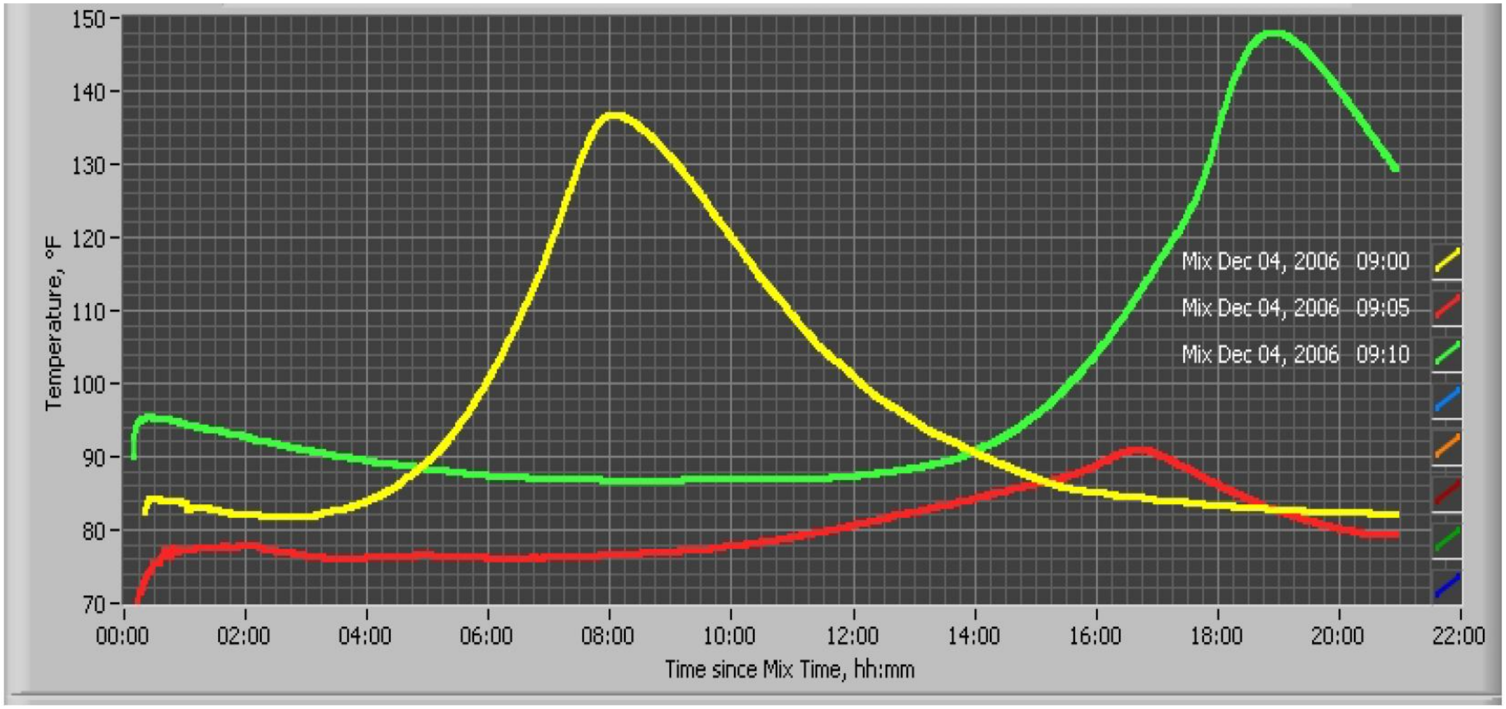
Other Miscellaneous reasons:

- **Time of addition**: [Hanehara & Yamada,1999], [Irshad & Agarwal,1994]
  - Increased workability when admixture addition delayed
  - However, delayed addition resulted in retarded set

Example: Compatibility of materials used



All mixes are 75% OPC, 25% class F fly ash

The green mix contains 3 oz/cwt of mid range water reducer, brand 1, plus single dose retarder

The red mix contains 3 oz/cwt of mid range water reducer, brand 2, plus single dose retarder

It appears that the brand 2 water reducer is not compatible with the combination of materials used. Little to no hydration occurs in the first 24 hours, indicating strong retardation and very little early strength gain.

GRACE

Courtesy of Darrick McGuire, Technical Specialist, WR Grace

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Other Miscellaneous reasons: ..

- **Increased Dosage**:

  [Aitcin et al., 1994], [Plante et al.,1989]

  – Higher dosage of water-reducing admixtures may also affect strength development

  – In some cases, severe slump loss has been encountered

  – Negatively influenced air void system

Other Miscellaneous reasons: …

- **Effect of temperature**:
  - Requires higher dosage of WRAs
  - Rapid slump loss
  - significant loss of air content [Dodson, 1990]
  - Strength loss

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Origins of Incompatibility (summary)

Early stiffening and workability	Retardation	Air content and stability
C_3A content	Low temperatures	Presence of carbon in fly ash
Sulfate content	Increased addition of plasticizer	Low alkali cements
High (>1%) and Low (<0.5%) alkalis	Interaction between admixtures	Type of air entrainer
Hot weather conditions	Delayed addition of plasticizer	Hot weather
Presence of lignin based admixtures		Interactions with water reducing admixtures
Timing of admixture addition		Timing of admixture addition

Previous Studies

- FHWA-HRT-06-079, (2006), Identifying Incompatible Combinations of Concrete Materials: Volume I–Final Report”
- FHWA-HRT-06-080, (2006), Identifying Incompatible Combinations of Concrete Materials: Volume II–Test Protocol”
- FHWA HIF-07-004 Report (2006), Integrated materials and Construction Practices for Concrete Pavement: A State-of-the-Practice Manual”

Test Methods Proposed to Study Incompatible Problems - Literature

Monitor Chemistry of Reactive Materials

Cement: C_3A and alkali content, fineness • SCM: calcium and alkali content, LOI
Chemical admixtures: composition

Stiffening	Cracking	Air Void System
Select Tests: • Slump loss. • Setting time. • Temperature development.	**Select Tests:** • HIPERPAV®. • Temperature development.	**Select Tests:** • Foam index. • Foam drainage. • Air content. • AVA.

Significant changes noted or problems occur?
Institute actions developed during preconstruction tests.
If problems continue, refer back to the laboratory.

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Outline

- Objectives.
- Literature Review
- Details of Experiment.
  - **Materials used for testing**
  - Work Plan.
  - Test Methods and Limiting Criteria for Incompatibility.
- Results.
- Future Work
- Conclusions

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Selection of Materials Used for Testing

- Materials for this study were selected from the list of INDOT approved materials (except for one fly ash high LOI used to study incompatibility problems related to air void system)

- Based on the literature review, materials which have a potential to generate incompatibility problems were selected (except for one cement).

Selection of Materials Used for Testing

- Following are the type of materials selected:
  - Four cements – varying chemical composition
  - Three fly ashes – varying chemical composition
    - Two class C fly ashes
    - One class F ash
  - Four plasticizers
  - Two air entraining agents

Properties of the Materials Selected for Testing

Chemical properties	Cements			
	C1	C2	C3	C4
C_3A %	9 (M)	10(M)	10.1(M)	7.7(L)
SO_3 %	3.0(M)	2.4(L)	3.6(H)	3.6(H)
Na_2O_{equ} %	0.29(L)	0.3(L)	1.04(H)	0.97(H)

Properties	Fly ashes		
	F1 Class C ash	F2 Class C ash	F3 Class F ash
LOI	0.38 (M)	0.25(L)	3.89
SO3	0.53(M)	1.14(H)	0.69(M)
Na_2O_{equ}	2.18(M)	1.94(M)	2.21(M)

*L – Low , M – Medium, H – High

Chemical Admixtures

- Water Reducing Admixtures: (WRA)
  - Lignin based Type A WRA (W1)
  - Polycarboxylate Type F superplasticizer (W2)
  - Lignin based Type E accelerating WRA (W3)
  - Lignin based Type D Retarding WRA (W4)
- Air Entraining Admixtures: (AEA)
  - Synthetic AEA (A1)
  - Vinsol resin (VR) based AEA (A2)

Nomenclature of Mixes

- Typical mix name : $C_iF_jW_kA_l$
  - C – cement
  - F -fly ash respectively
  - W - water reducing admixture
  - A air entraining admixture
  - ‘i, j, k, l’ stands for the variation within the specific type of material
- **Example:** C3F1W2 stands for a mix made with C3 type cement, Class C fly ash and PC type superplasticizer and the mix doesn’t have any air entraining agent.

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Work Plan

- **Phase I:** To identify incompatible mixes exhibiting early stiffening and abnormal setting behaviors.
  - Identify incompatible mixes by testing at room temperature and single dosage.
  - Evaluate the effect of the following on workability on limited number of mixes.
    - Double dosage WRAs
    - Delayed addition
    - Effect of Temperature

Work Plan …

- **Phase II:** To identify incompatible combinations w.r.t air void system

- **Phase III:** Validate the results on selected concrete mix

Experimental Plan

Phase I

Sub Task I

To identify incompatible combinations

•All combination of materials selected were studied

•**Cements - 4**	•**Class C ashes – 2**
•**2 WRA – Type A & Type F**	•**Synthetic &VR based AEA**

•Total 70 different combinations were studied

•Tests performed at 23°C and single dosage of WRA

•**Tests on pastes – Mini slump, Vicat's setting time**

•**Tests on mortars – Early age stiffening test, Semi adiabatic calorimetry**

Sub Task II

To study the effect of delayed addition of admixtures

- 3 each of retarding and normal mixes from sub task I

To study the effect of extra dosage of plasticizer

•3 each of normal and early stiffening mixes from sub task I

Effect of Temperature: (10 °C, 23 °C & 32 °C)

•At 32 °C: 3 each of normal and early stiffening mixes from phase I

•At 10 °C: 3 samples each of normal and slow stiffening mixes from phase I

Test methods – Mini slump, Early stiffening test, Vicat's set time

Plan of experimentation

Phase II

To study problems related to air void system

Materials:

- Low alkali cement (C1)
- Class F ash (F3)
- Type A (W1)and Type F (W2) WRAs
- Synthetic (A1) and VR based (A2) AEAs

Tests on paste : Foam Index, foam drainage.

Tests on mortar: Air content measurement (ASTM C 185)

Plan of experimentation …

Phase III

To validate the results from Task I & II on concrete:

Five most incompatible mixes from the all the above phases are selected for testing on concrete

Tests- Slump & air content at 10, 30 and 60 minutes , unit weight, hardened content air, compressive strength, semi adiabatic calorimetry

Outline

- Objectives.
- Literature Review.
- Details of Experiment.
  - Materials used for testing.
  - Work Plan.
  - **Test Methods and Limiting Criteria for Incompatibility**.
- Results.
- Future Work.
- Conclusions.

Test Methods Used to Study Incompatibility Problems

Reactive Materials:

Cement: C_3A , sulfate & alkali content   SCMs: sulfate% & LOI

WRAs & AEAs : Varying chemical nature

Stiffening:

- Early age stiffening (ASTM C 359)
- Vicat's Set time
- Mini Slump Testing
- Semi adiabatic calorimetry

Air Void System:

- Air content in mortars (ASTM C 185)
- Foam index testing
- Foam drainage testing

Validate the results on concrete mixes:

Slump loss	Unit weight	Compressive Strength
Loss in air content	Semi adiabatic Calorimetry	Hardened air content (ASTM C 457)

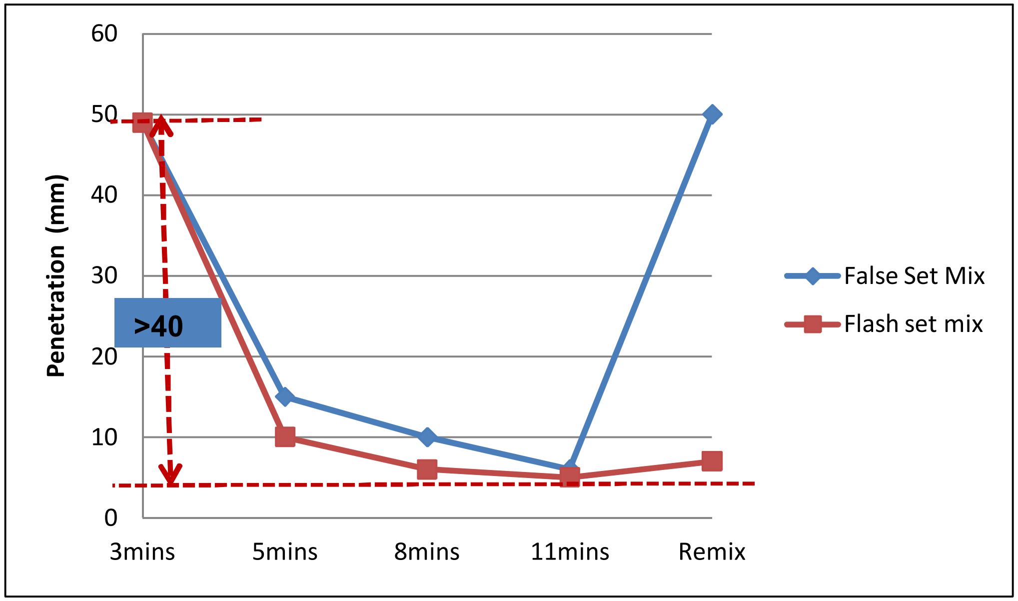
Early Age Stiffening Test (ASTM C 359):

- Performed on standard mortars made with graded and 20-30 standard sand.
- Standard consistency allowing plunger penetration of 46+/- 3 mm
- Mortar temperature after mixing is maintained at 23 (+/-)2 C
- Penetration at 3, 5, 8, 11minutes and after remixing were measured.

Limiting Criteria:

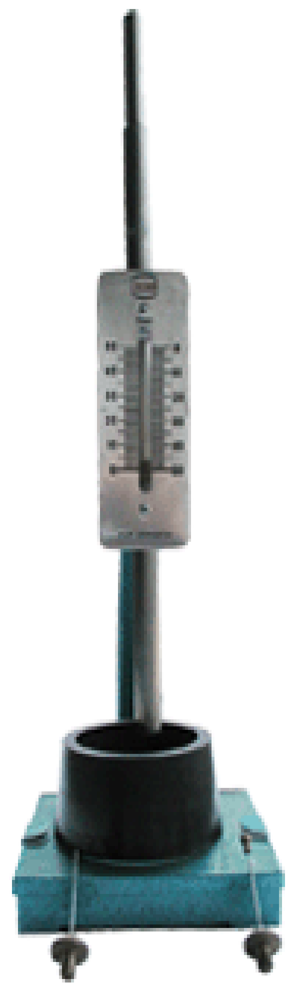
•Difference between 3 & 11 min penetrations > 40mm ➔ false set

•Remix penetration < 10 mm ➔ flash set



Set time Measurements using Vicat Apparatus (ASTM C 191)

- Performed on standard pastes made at normal consistency.
- Limiting Criteria:
  - Change in initial set time of 60 mins w.r.t base mix indicate potential incompatibility



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Semi-Adiabatic Calorimetry:

- Performed on mortars with W/C ratio 0.43
- Volume of paste in all the mixes is constant (400ml theoretically)
- Mixed according to ASTM C 305

Limiting Criteria:

- Changes in timing of max. temp peak > 60 mins w.r.t. base mix.
- Change in max. peak temperature > 6C (10F)
- Development of secondary peaks

Mini slump testing

- Performed on pastes - 0.43 w/c
- Weight of binder = 600gms
- High shear mixer (11000rpm) used
- Diameter of the spread measured at 2 , 5, 15, 30 and 45 minutes.

Limiting Criteria:

- F.S.I. ($Area_{5min}$/ $Area_{2min}$)>1.3 indicates potential false setting behavior.
- S.I. ($Area_{30min}$/ $Area_{5min}$)< 0.85 implies potential early stiffening mix.

Air content in Mortars (ASTM C 185)

- Performed on a low alkali cement (C1) and class F ash (F3) combinations.
- Performed on mortars (of standard flow 87.5 +/-7.5%) according to ASTM C 185
- Used to determine the dosage the dosage of AEA to achieve 18 +/- 2% air content.

Limiting Criteria:

- 20% change in the requirement of AEA dosage w.r.t. base mix indicate incompatible combinations.

Foam Index Testing

- Performed on slurries – 50ml water & 20g binder.
- Binder and water suspension were shaken in a 500 ml jar for 15 secs – homogeneous mixture.
- AEA was added and shaken for additional 15s.
- Used to determine the dosage of AEA needed for stable foam on top of liquid surface.

Limiting Criteria:

- 30% change in the requirement of AEA dosage w.r.t. base mix indicate incompatible combinations

Outline

- Objectives.
- Literature Review.
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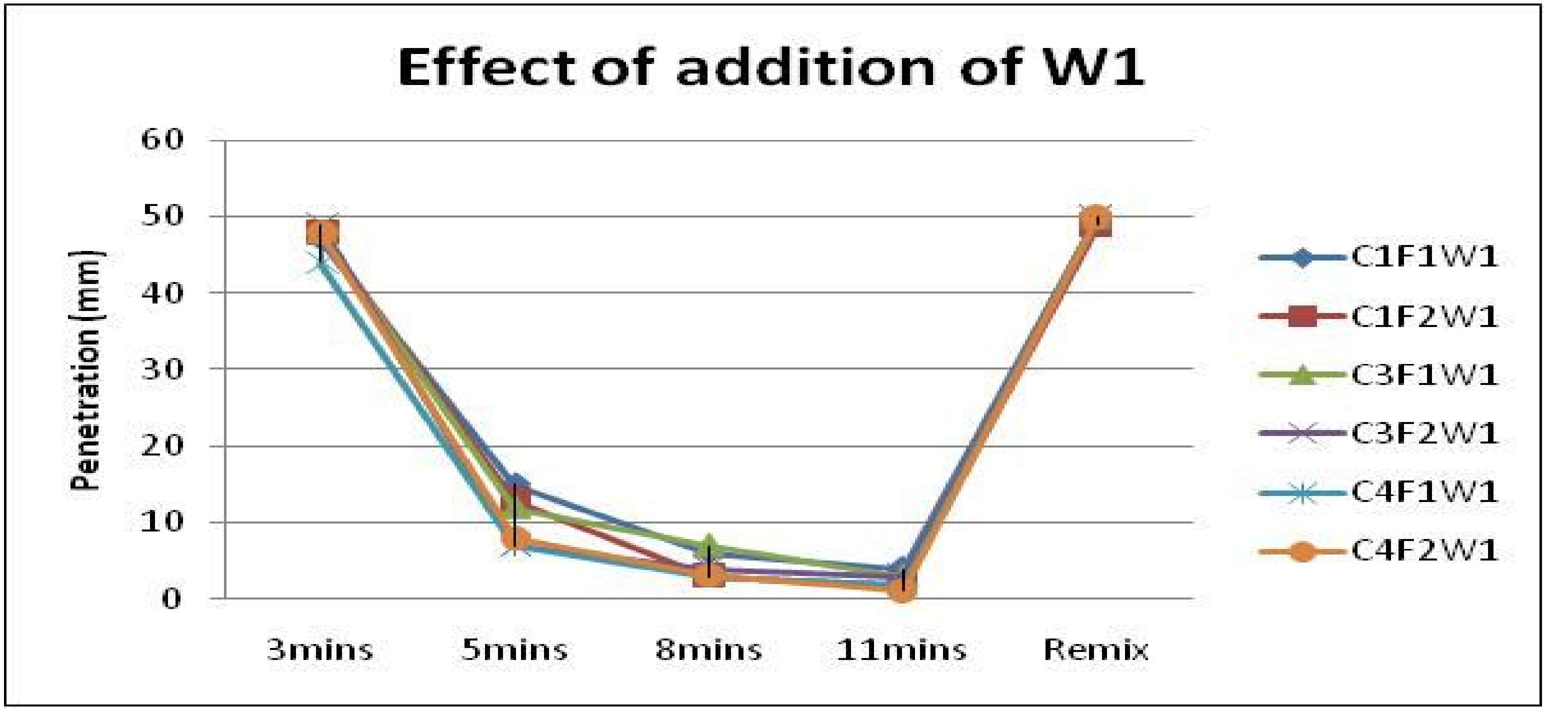
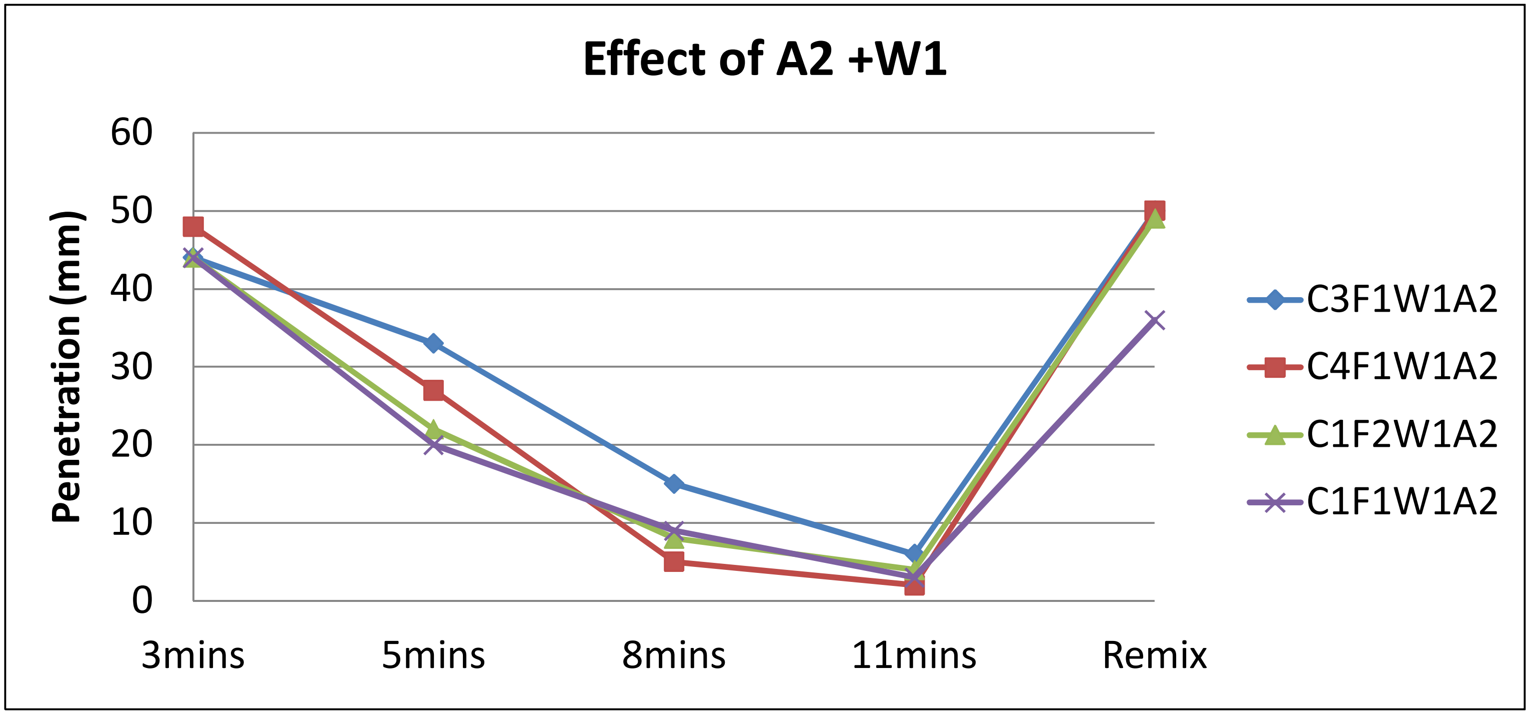
Early Age Stiffening Results

- Effect of WRAs:
  - W1- High sulfate (>3.1%) cements - False set
  - W2 – mixes with C1 or C2 – False set



Early Age Stiffening Results …contd

- Effect of AEAs in C+FA+WRS systems:
  - A1 or A2 + W2 –> mixes with C1 or C2 – false set
  - A2 + W1 –> mixes with high sulfate (>3.1%) cements (C1, C3, C4)– false set



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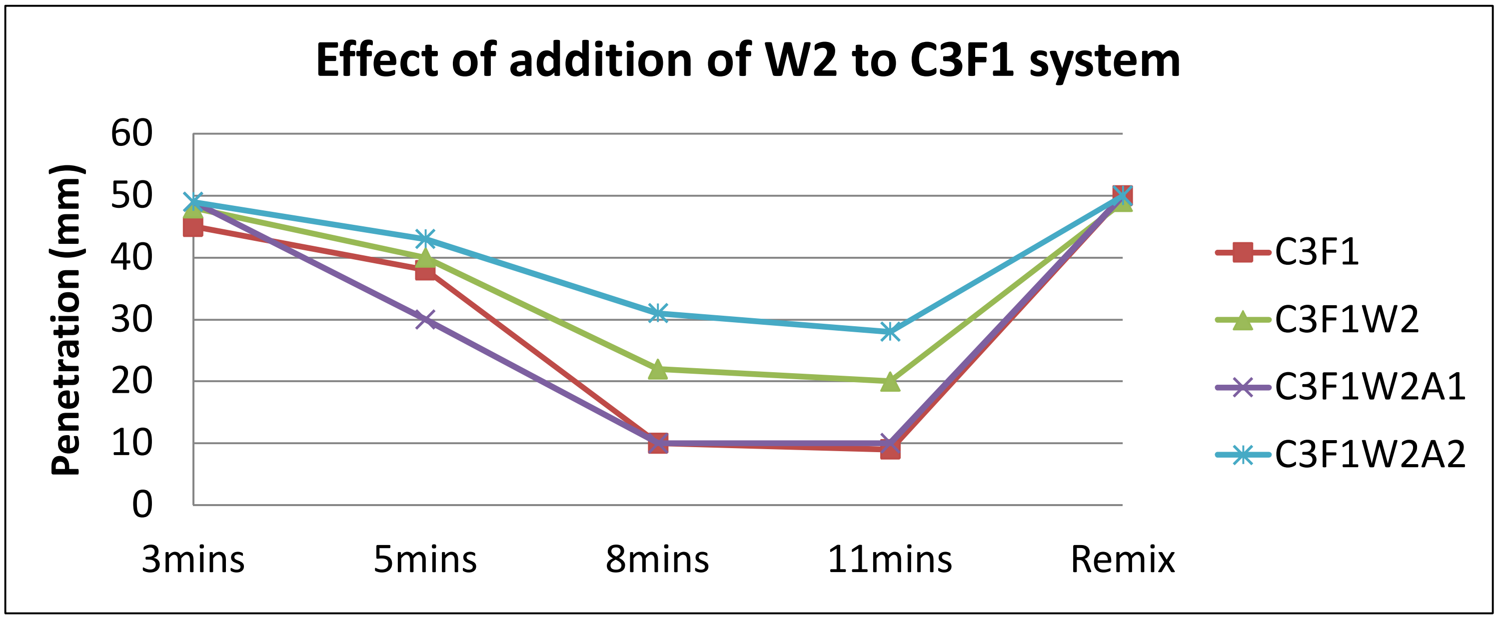
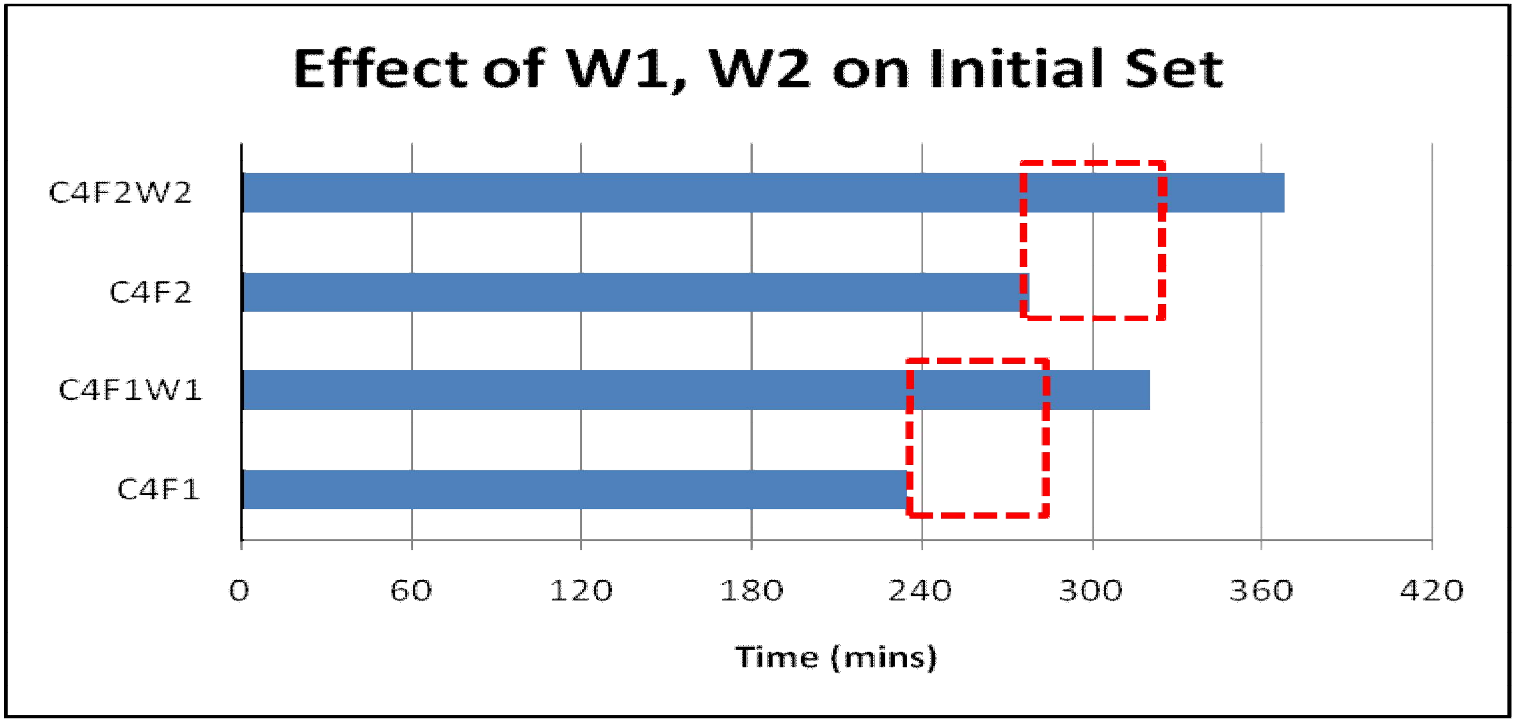
Early Stiffening Test – Compatible Combinations

- W2 – alone or with A1 or A2 - C3F1 or C3F2 system – no problem
- W1 + A1 or A2 to C3F1 – no problem
- W2 – alone or with A1 or A2 to C4F2 – no problem



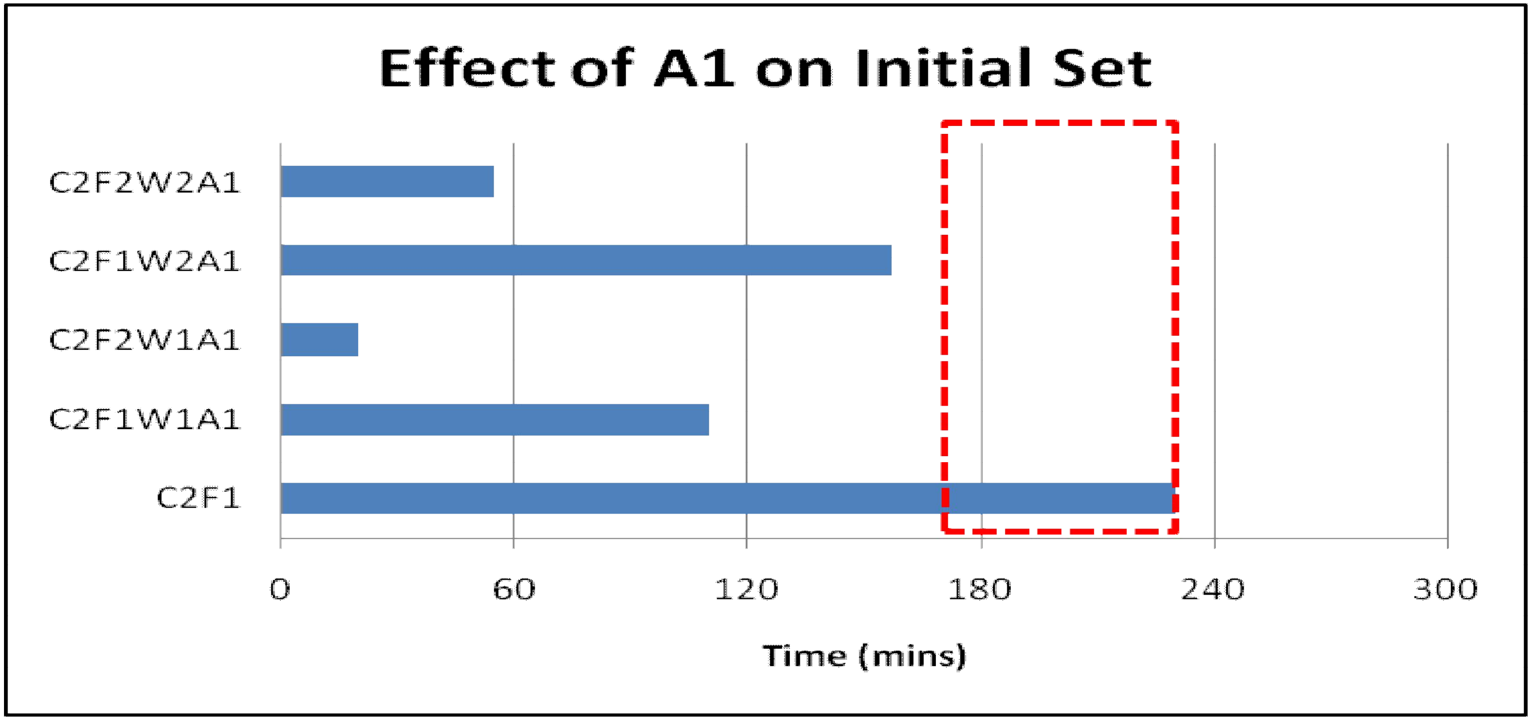
Vicat's Initial Set Time

- Effect of Plasticizers (W1, W2):
  - W1&W2 - Rapid acceleration of set with C2
  - W1&W2 - Severe retardation of set with C4



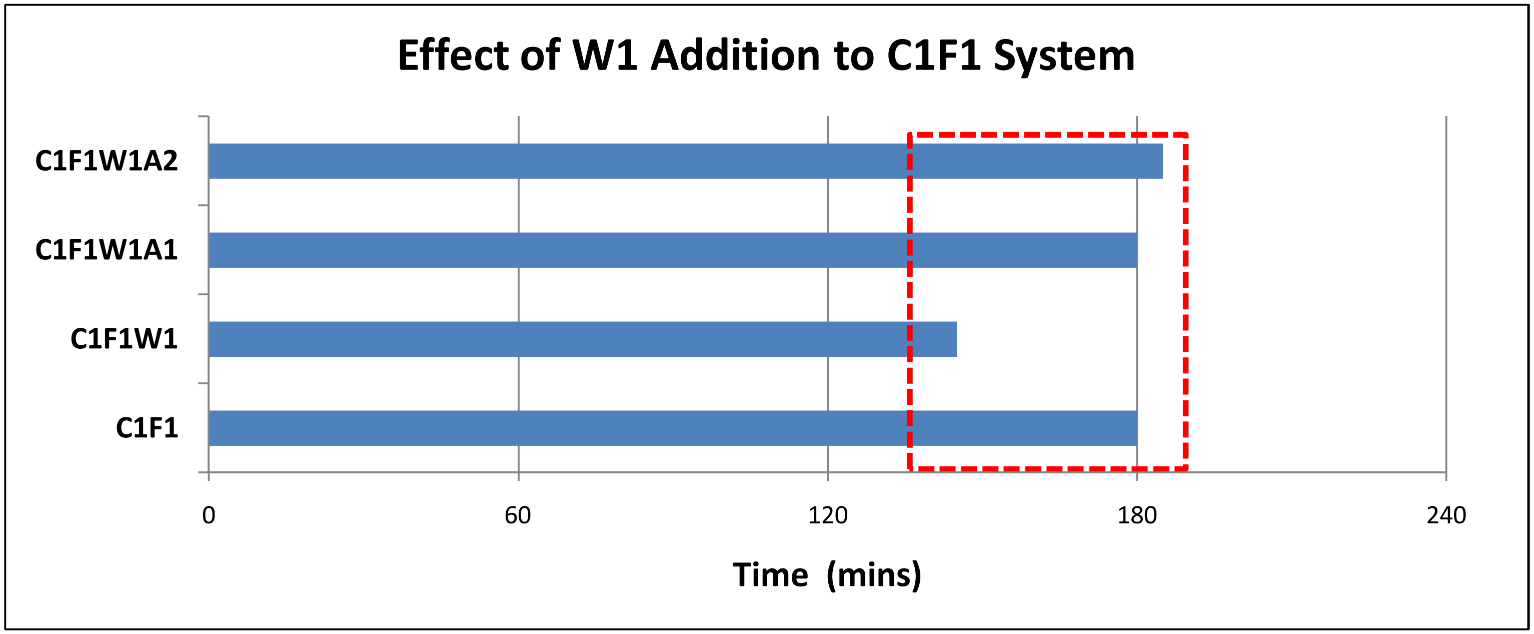
Vicat's Initial Set Time

- Effect of AEAs used with W1 or W2:
  - W1 & W2- Both AEA – rapid set with C2
  - W2- Both AEA – delayed set with C3 & F1



Set Time Experiment – Compatible Combinations

- C3F1 & W1 – alone or with AEAs – no problem
- C1F1 + W1 – alone with AEAs – no problem
- C3F2 + W2 – alone or with AEAs – no problem
- C1F2 + W2 – alone or with AEAs – no problem



Semi Adiabatic Calorimetry

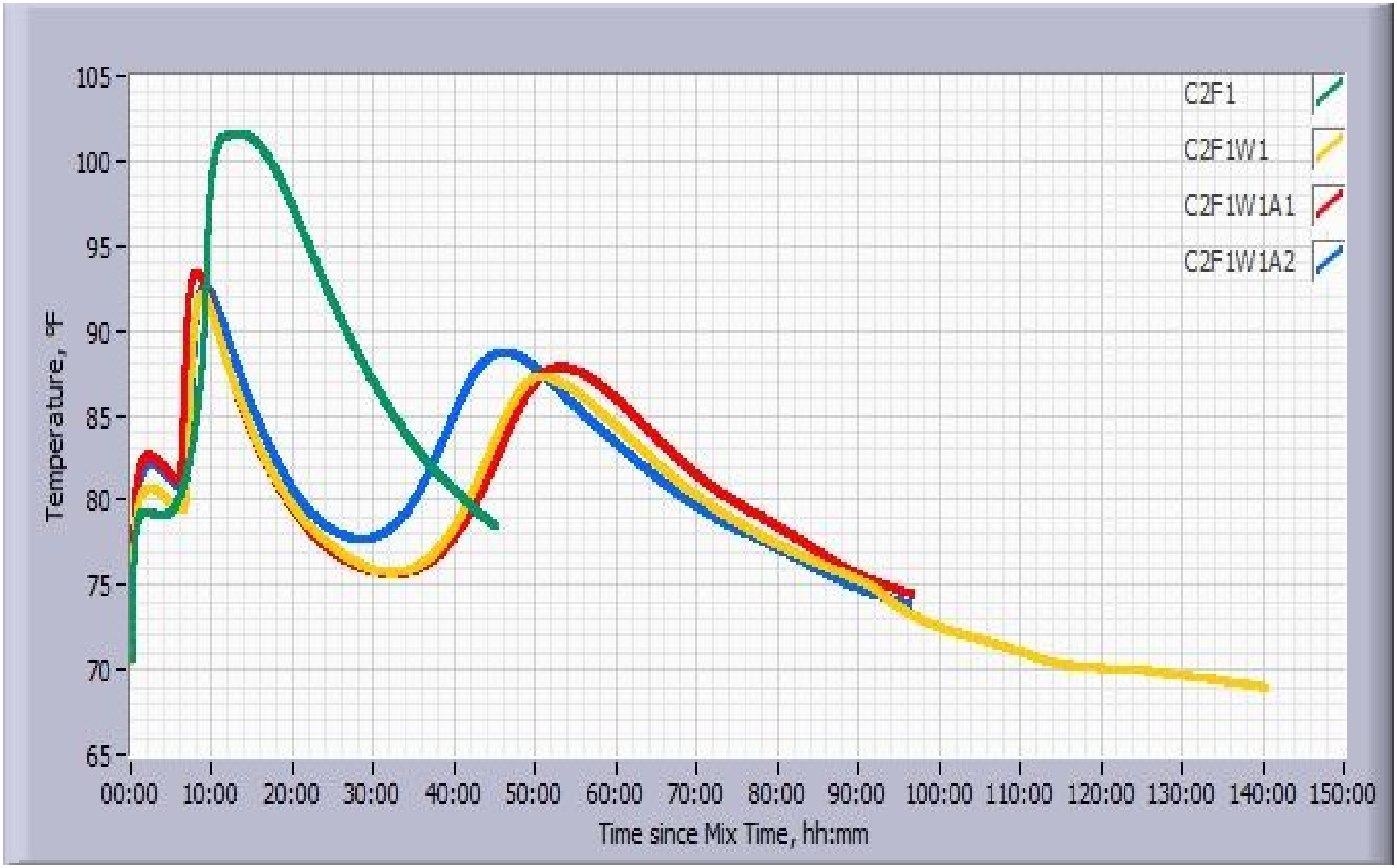
- W1 - C4F1 or C4F2 system
  - alone or with either A1 or A2
- W1 – C3F1 or C3F2 system
  - alone or with either A1 or A2
- W2 to C2+F1 system - alone or with either A1 or A2

Significant delayed the occurrence of max. peak temperature.

- W1 to C2 fly ash cementitious system - alone or along with A1 or A2

Accelerated the occurrence of peak and developed secondary peaks

Effect of W1 Addition to C2F1 System



Semi adiabatic Calorimetry- Compatible Combinations

- Replacing W1 with W2 in mixes containing C2 – no secondary peaks as seen in W1 + C2 + class C ash (F1 or F2) system.
- W2 addition – alone or with A1 or A2- C3 fly ash cementitious system – no problem
- W2 – alone or with A1 or A2 - C4F2 and C1F1 systems– no problem

Mini Slump Testing

- W1– alone or with A1 or A2 - C1+F2 systems resulted in early stiffening behavior
- W1 or W2 - alone or along with A2 - C2 systems - early stiffening behavior
- W1 or W2 – alone or with A2 - C3F2– false setting
- W1 - alone or with A1 or A2 – C4F1– early stiffening behavior

Mini slump Testing – Compatible Combinations

- Addition of W1 to C3F1 – alone or with A1 or A2 – did not exhibit any incompatibility problems
- Addition of W2 to C4 system (containing either of the fly ashes F1 or F2) – alone or along with A1 or A2 – significantly reduced the number of potentially incompatible mixes compared to that of mixes with other cements.

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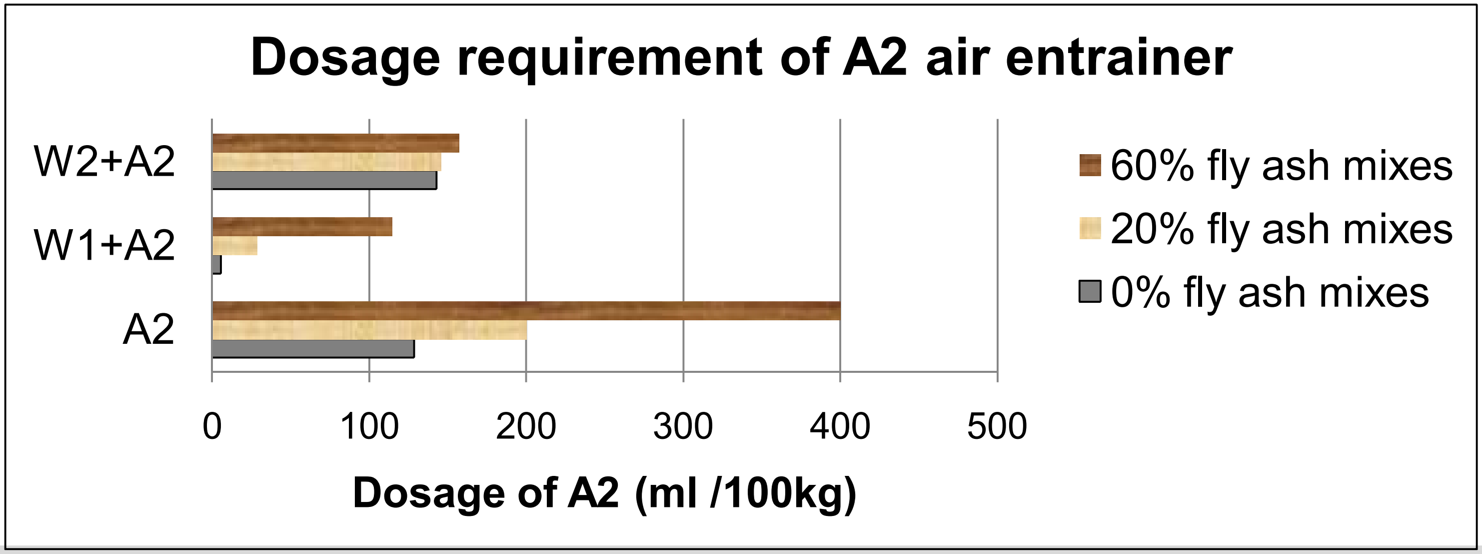
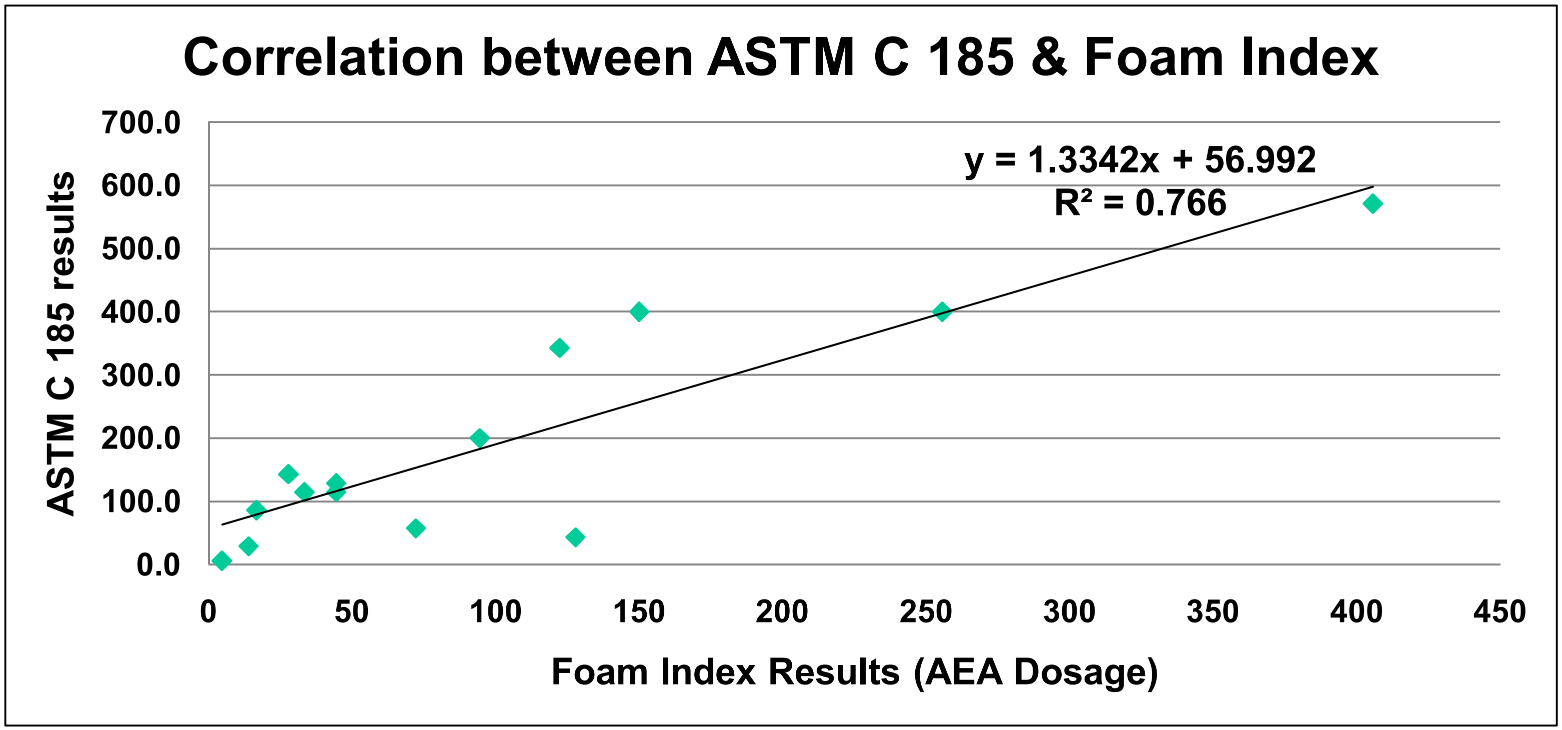
Air Content in mortars

- Addition of WRAs along with A1 or A2 reduced AEA dosage required to attain 18+/-2 % air content
- As the fly ash content increased, the amount of A1 or A2 (required to attain 18+/-2 % air content) increased except for mixes with W2
- In general, W1 had higher air entraining effect compared to that of W2



Foam Index Testing

- Foam index testing – Similar trends were observed as in ASTM C 185
- Reasonably good correlation between results of foam index testing and ASTM C 185 – R^2 value 0.77



Outline

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Future work

- Perform foam drainage testing
- Study the effect of temperature, delayed addition and dosage of admixtures on incompatibility
- Validate the results obtained from mortars and pastes on concrete mixes

Steps to Mitigate Incompatibility

Potential incompatible combination ???

Vary the following (one or more at a time) and test again for potential problems.

Stiffening:

- Select a cement with different chemical properties.
- Change the fly ash source
- Change the type of water reducing agent (WRA)
- Change the type of air entraining agent (AEA)

Air Void System:

- Select a fly ash with low LOI
- Change the percentage of fly ash replacement
- Change the type of air entraining agent (AEA)
- Change the type of Water reducing agent (WRA)

Test the new combination for incompatibility

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Scope for Future Work

- Expand the test matrix to accommodate different admixtures
- Expand the test matrix to study the effect of
  - increased fly ash replacement
  - other supplementary cementitious materials

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Conclusions

- Addition of poly carboxylate type F WRA (W2) to high C_3A (>=9%) & low alkali (<=0.7% total) content fly ash cementitious systems (C1,C2) resulted in stiffening related problems.
- When used with W1, high C_3A (>9%) & low alkali (<=0.7% total) content fly ash cementitious systems (C1,C2), VR based(A2) AEAs, resulted in abnormal stiffening behavior.
- Both W1 & W2 when used with Low (<8%) C_3A content and high (>3.1%) sulfate content fly ash cementitious systems (C4) resulted in severe retardation of set.

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Conclusions …contd

- Addition of W1 or W2 to low (<2.8 %) total sulfate content fly ash cementitious systems (C2) resulted in rapid acceleration of set .
- Addition of AEAS along with W2 to C2 (high C_3A =10% low total alkali (<0.7%) and low total sulfate (<2.8%)) cementitious system resulted in significant decrease in initial set time.
- AEA dosage requirement for mixes containing W1 was found to be more sensitive to the dosage of the fly ash compared to that of mixes with W2.

Conclusions ...contd

- Addition of W1 to C2 + class C ash system, resulted in development of secondary peaks indicating significant changes to hydration process.
- One should be aware of possibility of incompatibility problems and perform tests to confirm compatibility whenever:
  - Change in Cement or SCM source.
  - Change in the type of admixture.
  - Time of addition and/ or amount of admixture varied.
  - Temperature varies.

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References:

- Aitcin, P.C., Jolicoeur, C, and MacGregor, J.G., “Superplasticizers: How They Work and Why They Occasionally Don’t,” *Concrete International, Vol. 16, No. 5, May 1994,* pp. 45–52.
- Ansari .F, Zhijun Zhang, Ali Maher and Patrick Szary “Effect of synthetic air entraining agents on compressive strength of Portland cement concrete mechanism of interaction and remediation strategy” *FHWA*, 2002. NJ-2002-025
- Claude Bedard P.E. , Noel Mailvaganam, “The use of Chemical Admixtures in concrete. Part II: Admixture-Admixture compatibility and Practical Problems” , *journal of Performance of Constructed Facilities , ASCE*, Feb 2006
- Cross, W., Duke, E., Kellar, J., Johnson, D. “Investigation of low compressive strengths of concrete paving, precast and structural concrete*” South Dakota department of transportation,* 2000, SD98-03-F
- Dodson. V.H., Concrete admixtures. New York : Van Nostrand Reinhold, 1990.
- Gebler, S.H., Klieger, P. Effect of fly ash on air void stability of concrete*. Skokie, Illinois : Portland Cement association*, 1983. RD085.01T.

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References ….

- Gress, D., Early Distress in Concrete Pavements, *Federal Highway Administration,*FHWA-SA-97-045, Washington, DC, January 9, 1997, pp. 53.
- Gao Y-M, Shim H-S, Hurt RH, Suuberg EM, Yang NYC, Effects of carbon on air entrainment in fly ash concrete: the role of soot and carbon black", *Energy Fuels* 1997;11(2):457–62.
- Hanehara.S, Yamada,Kazuo, "Interaction between cement and chemical admixtures "*Cem. Conc and Res*. 29(1999) 1159 – 1165
- Indrek K Laots, Robert H. Hurt, Eric M. Suuberg , "Size distribution of unburned carbon in coal fly ash and its implications*"* : *Fuel*, 2004, pp. 223-230.
- Irshad Masood and S.K. Agarwal, "Effects of various super plasticizers on rheological properties of cement and mortar", *Cem and Conc. Res*.(1994) 24, 291-302
- Jennings, H., Johnson, D.L., Moss, G.M., and Saak, A.W., "Pooled Fund Study of Premature Rigid Deterioration"*, Iowa Department of Transportation Research Project H-*1063, Northwestern University Technological Institute, Evanston, IL, April 1997, 94 pp.

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References….

- Paul J. Sandberg and Lawrence R. Roberts, "Cement – Admixture Interaction Related to Aluminate control", *Journal of ASTM International*, June 2005, Vol2 No 6
- Plante, P., Pigeon, M. and Foy, C. "The Influence of Water-Reducers on the Production and Stability of the Air void System in Concrete," *Cem. and Conc Res., Vol.*19, No. 4, 1989, pp. 621–633
- Prince .W., M. Espagne, P.C. Aitcin, "Ettringite formation : crucial step in cement super plasticizer compatibility" *Cem.Conc and Res.* 33(2002) 635- 641
- Shiping Jian, Byung Gi Kim Pierre-Claude Aitcin, "Importance of adequate soluble alkalis to ensure cement/super plasticizer compatibility" *Cem. Conc Res.* 29(1999) 71 – 78
- Taylor. P.C, Luis A. Graf, Jerzy Z. Zemajtis, " Identifying incompatible combinations of concrete materials: Volume I – Final repot", *FHWA* Publication no. FHWA-HRT-06-079, Aug 2006
- Wang. H, C. Qi, Hamid Farzam, Jim Turici, "Interactions of materials used in cement: Effect of fly ash and admixtures on concrete ", *Concrete international* , April 2006
- Zhang, D.S. , "Air entrainment in pozzolanic fly ashes", *Cement and Concrete Composites,* 1996, pp. 409-416.