# ALKALI-SILICA REACTION AND DELAYED ETTRINGITE FORMATION IN RAPID SETTING HYDRAULIC CEMENTITIOUS SYSTEMS

by

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# **DEDICATION**

To my family.

## **ACKNOWLEDGMENTS**

There are so many people I would like to thank at this moment. I would like to start thanking my family for their support and for always encouraging me to chase my dreams and make them true. To my parents, Rosana and Francisco, you both are my motivation. I would like to thank Dr. Aguayo for giving me the opportunity of working with him on this project. This project would have not been possible without the help of my lab teammates. Thanks for the early mornings and the hard work. Lastly, I would like to thank my thesis committee for their guidance and mentoring throughout the development of this project.

# TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENTS	V
LIST OF TABLES	ix
LIST OF FIGURES	xi
LIST OF ABBREVIATIONS	xiv
ABSTRACT	XV
CHAPTER	
1. INTRODUCTION	1
1.1 Background 1.2 Statement of Problem 1.3 Research Significance 1.4 Objectives 1.5 Delimitations and limitations 1.6 Assumptions 1.7 Thesis organization	1 3 3
2. LITERATURE REVIEW	
2.1 Rapid Setting Hydraulic Cementitious Systems (RSHC)	7 9 10 10
2.1.3 Blended system incorporating CSA or CAC with OPC 2.1.3.1 Background and history	

	2.1.3.2 Cement chemistry and hydration of blended systems.	12
	2.1.3.3 Application of blended systems	
	2.2 Alkali-Silica Reaction (ASR)	
	2.2.1 Historical background	
	2.2.2 Mechanisms and essential components	
	2.2.3 Laboratory and field-testing methods	
	2.2.3.1 Accelerated Mortar Bar Test Method (ASTM C1260)	
	2.2.3.2 Concrete Prism Test (ASTM C1293)	
	2.2.3.3 Concrete Blocks	
	2.2.4 ASR performance of RSHC's	
	2.3 Delayed Ettringite Formation (DEF)	
	2.3.1 Historical background	
	2.3.2 Mechanisms and essential components	
	2.3.3 Testing methods	
	2.3.4 DEF performance of RSHC's	
	1	
3.	METHODOLOGY	27
	3.1 Experimental Program	27
	3.2 Materials and mixture proportions	27
	3.3 Alkali-silica reactivity experimental methods	
	3.3.1 Concrete exposure blocks	
	3.3.1.1 Materials and mixture proportions	
	3.3.1.2 Timeline and procedure	36
	3.3.2 Concrete Prism Test (ASTM C1293)	40
	3.3.2.1 Materials and mixture proportions	40
	3.3.2.2 Timeline and procedure	44
	3.3.3 Accelerated Mortar Bar Test (ASTM C1260/C1567)	45
	3.3.3.1 Materials and mixture proportions	45
	3.3.3.2 Timeline and procedure	47
	3.4 Delayed Ettringite Formation experimental methods	49
	3.4.1 Accelerated high temp curing (Kelham Test Method)	49
	3.4.1.1 Materials and mixture proportions	51
	3.4.1.2 Timeline and procedure	51
4.	RESULTS AND DISCUSSIONS	53
	4.1 Alkali silica reaction	
	4.1.1 Concrete exposure blocks	
	4.1.1.1 Hydration temp and relative humidity outcomes	
	4.1.2 Concrete Prisms Test (ASTM C1293)	60

4.1.3 Accelerated Mortar Bar Test (ASTM C1260)	79
4.2 Delayed ettringite formation	91
4.2.1 Accelerated High Temp Curing (Kelham Test Method)	
5. CONCLUSIONS AND RECOMMENDATIONS	96
5.1 Durability performance	96
5.2 Future work	99
REFERENCES	101

# LIST OF TABLES

Ta	Page
1.	Chemical Properties of OPC and CAC
2.	Comparison between OPC and CSA cements
3.	Mortar Bar Expansion Values
4.	CPT Expansion Values
5.	Aggregates:Types, Categories and Origins
6.	Cements: Blend Types, Tags and Types
7.	Cements: Chemical Properties
8.	Admixtures: Description and Potential Outcomes
9.	Concrete Exposure Blocks: Mixture Design
10.	CPT: Series 1 Undoped Mixture Design
11.	CPT: Series 2 Doped Mixture Design
12.	CPT: Series 3 Doped Mixture Design
13.	Sieving Grades for El Indio, Limestone and Jobe
14.	Fine Aggregate Grading

15. Mortar Bar Test ASTM C1260: Mixture Design	47
16. DEF Series 1 and 2: Mixture Design	51
17. Concrete Exposure Blocks: Average Expansion Days 28 through 183	53
18. Concrete Exposure Blocks: CSA-B1 and CSA-B2 Chemical Composition	58
19. Concrete Exposure Blocks: Min and Max Temperatures in Fahrenheit	63
20. CPT: Series 1 Expansion Averages Days 28 through 183	69
21. CPT: Series 2 Expansion Averages Days 28 through 183	72
22. CPT: Series 3 Expansion Averages Days 28 through 183	75
23. DEF – Kelham Method 95F Expansion Rates Day 0 through 183	92
24. DEF – Modified Method 65F Expansion Rates Day 0 through 183	94

# LIST OF FIGURES

Fig	gure Pa	age
1.	Thomas E. Stanton showing structure affected by ASR	. 14
2.	ASR components	. 15
3.	ASR Mechanism	. 16
4.	Concrete Structures Affected by ASR	. 18
5.	Concrete Structures Affected by DEF	. 22
6.	DEF Components	. 23
7.	DEF Mechanism	. 24
8.	Exposure Concrete Block Site at Texas State University – February 15, 2021	. 34
9.	Exterior View of 16 x 16 x 16 in Wooden Mold.	. 36
10.	. Gauge Measurement Device	. 37
11.	. Interior View of 16 x 16 x 16 in Wooden Mold	. 38
12.	. Completed Concrete Exposure Block – CSA2	. 39
13.	. Container Storing Prims ASTM C1293	. 44
14.	. Mortar Bars Submerged in NaOH Solution and Measurement Apparatus	. 48

15. Series 1: Kelham Method for DEF Curing	50
16. Series 2: Modified Method for DEF Curing	50
17. Overall Expansion Rates for CSA, CAC, and OPC in Exposure Blocks	54
18. CAC System Expansion Rates	55
19. CAC1 Concrete Block 5 Sides	56
20. CAC1 Physical Damages on Side 5	56
21. CSA System Expansion Rates	57
22. CSA-B1 Concrete Exposure Block	58
23. CSA-B2 Concrete Exposure Block	59
24. Blended System Expansion Rates	60
25. CSA-OPC2 Concrete Exposure Blocks	61
26. CAC-OPC2 Concrete Exposure Blocks	62
27. CAC1 - First 7 Days Temperature Report Hilti Sensor	64
28. CAC1 - First 28 Days Relative Humidity Report Hilti Sensor	65
29. CAC-B1 - PP5001 First 7 Days Temperature Report Hilti Sensor	67
30. CAC-B1 - PP5001 First 28 Days Relative Humidity Report Hilti Sensor	68

31. CPT Series 1 – Overall Expansion Rates for CSA, CAC, and OPC	70
32. CPT Series 1 – Expansion Less than 0.04%	71
33. CPT Series 2 – Overall Expansion Rates for CSA, CAC and OPC	73
34. Series 2 Binders Tending to Pass CPT Under 0.04%	74
35. CPT Series 3 – Overall Expansion Rates for CSA, CAC and OPC	76
36. CPT Series 3 – Expansion Less than 0.04%	77
37. CSA1 25% - OPC2 75% (2) Cracks on CSA1 25% Specimen B	78
38. CSA1 100% Specimen B on Measurement Apparatuss	78
39. OPC – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion	80
40. CAC – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion	82
41. CSA – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion	84
42. CAC/OPC2 blends – Limestone, El Indio, Jobe Overall Expansion	87
43. CSA/OPC2 blends – Limestone, El Indio, Jobe Overall Expansion	89
44. (1) El Indio, CSA1 75% - OPC2 25% prisms	89
45. Kelham Method 95°F Peak Series Expansion with Time	93
46. Modified Method 65°F Peak Series Expansion with Time	95

## LIST OF ABBREVIATIONS

**Abbreviation Description** 

(W/CM) Water-Cement Ratio

AAC Alkali-Activated Cement

AH3 Alumina Gel

ASR Alkali-Silica Reaction

ASTM American Society for Testing Materials

CA Monocalcium Aluminate

CAC Calcium Aluminate Cement

CPT Concrete Prism Test

CSA Calcium Sulfoaluminate Cement

DEF Delayed Ettringite Formation

OPC Ordinary Portland Cement

PSI Per Square Inch

RH Relative Humidity

RSHC Rapid Setting Hydraulic Cementitious System

#### **ABSTRACT**

The objective of this thesis is to generate a deeper understanding of how rapid setting and hardening cements perform when being exposed to environments that can hypothetically accelerate or reduce the alkali-silica reaction and delayed ettringite formation. It has been proved in preceding literature studies that ordinary portland cements expands to both coarse and fine aggregates depending on their level of known reactivity. In order to generate precise outcomes for the study of both Calcium Aluminate and Calcium Sulfoaluminate Cements' reactivity, coarse and fine aggregates identified as non-reactive were utilized. Moreover, concrete exposure blocks were analyzed while exposed to field conditions, and concrete prisms and mortar bars were studied in controlled laboratory conditions. Specimens were studied based on standard and modified testing methods such as ASTM C1293, ASTM C1260, and the Kelham Method.

#### 1. INTRODUCTION

# 1.1 Background

In collaboration with the Texas Department of Transportation (TxDOT) and the Department of Engineering Technology at Texas State University, this study took place to deliver a more profound understanding of the effects of Alkali-Silica Reaction (ASR) and Delayed Ettringite Formation (DEF) in rapid setting and hardening cement concrete. The significance of this project related to the negative consequences that both ASR and DEF reactions generate to Texas transportation infrastructure.

#### 1.2 Statement of Problem

There have been some major noticeable damages in the form of cracks during the past few years in some Texas concrete infrastructure such as bridges and roadways. Many of these noticeable cracks have been extremely concerning and have been found to be caused by ASR and DEF (Thomas, 2013). ASR is a chemical reaction between highly alkaline cement paste and the reactive non-crystalline silica found in aggregates added to cement concrete mixtures. This reaction also involves various types of reactive silica (SiO<sub>2</sub>) minerals (Fournier, 2010). ASR produces a swelling response that arises over time causing the concrete to crack and is characterized by being an expansive reaction that leads to a reduction in the service life of most concrete structures (Giannini, 2013). DEF is the formation of ettringite in moist concrete, or mortar after the destruction of primary ettringite when exposed to high temperatures. DEF is also characterized for causing damages to materials and structures bared at temperatures higher than 70°C (Taylor, 2000).

According to the TxDOT, transportation structures in Texas are affected by a magnitude due to the warm and humid weather that characterizes the area. It is concerning that these ASR and DEF deterioration damages are valued to be roughly about \$1 billion a year (Giannini, 2013). Therefore, evaluating and managing the effects on ASR and DEF and the possible prevention by using rapid setting cement would seem economically feasible and important for new construction.

Although rapid-hardening cements are not as durable as ordinary portland cement, they are a great solution to meet the schedule requirements and budget needs in a project. While OPC has been successfully used for many years in the construction industry, it is more prone to drying shrinkage and cracking (Trejo, 2017). The principal idea for this project is to prove the fact that when accelerating the strength gain in Calcium Sulfoaluminate (CSA) and Calcium Aluminate cements (CAC), the chemical additives reduce shrinkage and will have major resistance to ASR reactivity and DEF within the aggregates.

# 1.3 Research Significance

- To obtain short and long-term data for ASR and DEF of Calcium Aluminate, Calcium Sulfoaluminate, and blended binders systems containing CAC/OPC and CSA/OPC.
- 2. To generate data that will show how performance varies in these systems based on standard and modified testing methods.
- 3. To generate data that will define the quality and durability of RSHCs.

## 1.4 Objectives

- Understanding the reactivity of alkalis and sulfates of RSHCs with respect to ASR and DEF.
- 2. Understanding mechanisms within CAC, CSA, and blended binders with respect to ASR and DEF.
- Determining the influence of standardized and modified performance test methods on RSHCs related to ASR and DEF.
- 4. Providing sufficient short-term and long-term data to define the quality and durability of RSHCs.

#### 1.5 Delimitations and limitations

- The study will be limited to examining only the reactivity when using CAC and CSA cements.
- 2. The objective uses OPC as a means to discover new DEF reactions and expansive alkali-silica reactions in CAC and CSA cements.
- 3. The principal limitation for the development of this project is the shortage of information regarding fast setting cements. One of the reasons why the data about rapid setting cements is limited is because these cements are not as popular in the construction industry due to their cost.
- 4. The unpopularity of rapid setting cements affects the acquisition of materials for the development of this project. The construction industry still relies on affordability, durability, and reliability; therefore, it is more common for them to use OPC instead of RSHC's. Although RSHCs have been out in the industry for decades, these types are still seen as a higher advancement and

are not very demanded in the construction industry. Going further on obtaining data and information, another limitation turned out to be that there is not enough historic data information on ASR or DEF on RSHCs.

5. The research is delimited to historical findings, the collection of measurements, and its data analysis. This testing process can take up to two years to be completed for all the mixtures, therefore, consistency and availability to test each specimen must be carefully controlled.

# 1.6 Assumptions

- Binders managing reactive aggregates should start to show expansion due to ASR at 1 year.
- 2. Mixtures that were doped with additional alkali (NaOH) should show more and a quicker onset of expansion than those that were left as a control.
- 3. These binders used are known to exhibit rapid setting and hardening abilities due to the production of ettringite in their initial hours of hydration, consequently, the setting times are correlated to the amount of ettringite developed at curing times.

## 1.7 Thesis organization

In the direction of achieving the objectives, this report is divided into three chapters. The first chapter is the introduction which includes a background of the problem and research significance. The second chapter includes the literature review on Rapid Setting Hydraulic Cementitious Systems, experimental methods based on standard and modified laboratory test, and several field tests of samples. The third chapter includes an explanation of the methodology and the laboratory experimental program. The

laboratory program contains details about the development of the accelerated mortar bar test ASTM C1260, and the concrete prism test ASTM C1293 for ASR, and specimens evaluated for DEF will include subjecting samples to an accelerated high temperature curing regime based on the Kelham Method. These standards include specifications regarding mixture methods, specimen curing times, storage techniques, and temperature and dates for measurements to perceive ASR and DEF results. The fourth chapter of this report presents the results and discussion of both laboratory and field tests performed for the matter of the research goal. The fifth and final chapter includes the conclusion and future studies.

#### 2. LITERATURE REVIEW

# 2.1 Rapid Setting Hydraulic Cementitious Systems (RSHC)

Cement is a major component of concrete which constitutes about 15 to 25% of the binding phase in concrete (C.K.Y. Leung, 2009). When utilizing ordinary portland cement, there are more materials that are added to a concrete mixture for different purposes. These materials are called admixtures and are added to the mixture immediately before or during mixing (Design and Control of Concrete Mixtures).

The most popular type of cement used in the construction industry has been OPC. According to Zhang (2018), using Type I portland cement in structural concrete delivers the property to gain compressive strength between 3,000 and 6,000 psi in the first week to 10 days of curing (Zhang, 2018). Although the compressive strength of the mixture depends upon different aspects, the most important indicator of strength is the ratio of the water used compared to the amount of cement (Design and Control of Concrete Mixtures). Typically, when there is a lower water-to-cement ratio (w/c), there will be a higher compressive strength. In recent decades there has been an increased utilization of non-portland cements that can be more appropriate for determined applications. Rapid setting, high early strength, and durability are characteristics that define the type of alternate cement system needed.

Calcium Sulfoaluminate and Aluminate cements have been around the construction industry since the 1960s (Horr, 2017). These two types of cements are characterized for leading concrete mixtures to early strength development, and less shrinking when compared to OPC. The use of rapid setting cements is convenient for the

construction of concrete roads, bridges and the fast-track pavement rehabilitation of highways. CAC and CSA cements contain different components compared to OPC, which makes them an innovative, but uncommonly, used material.

According to the American Concrete Institute (PRC-225-19) "CAC and CSA cements are hydraulic cements that are obtained by pulverizing a solidified melt or clinker that consists of hydraulic calcium aluminates formed from proportioned mixtures of aluminous and calcareous materials". These components are divided into three groups that are based on alumina and iron oxide contents. The quantities of alumina and iron oxide define whether the material has low, intermediate, or high purity. Generally, cements that contain higher alumina content are suitable for higher temperature applications. CAC and CSA cements are characterized for containing less silica than OPCs (Tan, 2020). Therefore, CAC and CSA are high-early-strength and quick-setting characteristics (ACI).

Admixtures are typically classified depending on their function. Admixtures can be utilized as air-entraining, water-reducing, plasticizers, accelerating, retarding, hydration-control, corrosion, shrinkage, and as ASR reactivity inhibitors. Besides these functions, admixtures are used to decrease the cost of concrete construction, be more effective and maintain quality throughout all types of conditions.

#### **2.1.1** Calcium Aluminate Cements (CAC)

#### 2.1.1.1 Background and history

CACs are known for managing a rapid setting property while developing low temperatures. CACs have been introduced into the construction industry thanks to their

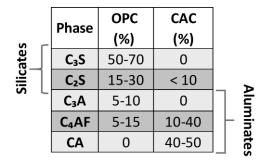
superior durability and high-temperature resistance. CACs are composed by nearly 40 percent of alumina, 40 percent of lime, 15 percent of ferric or ferrous oxides, and about 5 percent of fused silica that can also contain reduced amounts of titanium dioxide, silica, and magnesia (Kurtis et al, 1999). Additionally, bauxite and limestone are the raw materials major components to produce CAC. These major components led CACs to be known as high-alumina cements or cement fondu.

# 2.1.1.2 Cement chemistry and hydration of CAC.

CAC is principally composed of monocalcium aluminate (CA) which is about 50 to 60 percent of the cement by weight (Kurtis et al, 1999). The rapid setting characteristic of CAC is generated by the hydration of CA. The hydration of CA produces CAH<sub>10</sub>, condensed quantities of C<sub>2</sub>AH<sub>8</sub> as well as alumina gel (AH<sub>3</sub>). When exposed to higher temperatures, higher concentrations of lime and a rise in alkalinity could be generated (Rambo et al, 2019).

The hydration of CAC primarily consists of calcium aluminate CA, C<sub>12</sub>A<sub>7</sub>, and CA<sub>2</sub> (Zhang et al, 2018). C<sub>12</sub>A<sub>7</sub> becomes a vital component of CAC which is devoted to hydrate to C<sub>2</sub>AH<sub>8</sub>. Once time passes, either at elevated or normal temperatures, both components, CAH<sub>10</sub> and C<sub>2</sub>AH<sub>8</sub> convert into C<sub>3</sub>AH<sub>6</sub> and AH<sub>3</sub>.

Table 1. Chemical Properties of OPC and CAC (Kurtis et al, 1999)



Rapid setting and strength gaining concrete based on CAC is made using a high amount of superplasticizer which aids the maintenance of a low w/c ratio, as well as an accelerator with the aim to trigger early hardening conditions. The high early strength of CAC is credited to the type of accelerator added in (Rambo et al, 2019). The addition of accelerators and high range water reduction is vital to allow CAC to have an extended working time as the working time of this material is limited. Although lithium-based accelerators are the most used together with CAC, it is important to consult with the cement producer before preparing batch trials.

# **2.1.1.3** Application of CAC concrete

CAC is characterized for having a higher early strength gain and a high heat of hydration comparing to OPC (TxDOT, 2010). These characteristics allow the usage of CAC attractive in the construction industry especially during the winter months and/or when speedy repairs are needed. Designed to be a sulfate-resistant cement, CAC is an ideal tool for concrete pours in extreme cold weather conditions as CAC still allows the achievement of high early strength gains.

Zapata (2019) stated that calcium aluminate cements (CAC) are used in high-performance applications, as well as in situations that require resistance to chemical attacks, generate a high early strength and strong resistance. Zapata's study was based on investigating the behavior of CACs during high-temperature conditions. Although Zapata's study exposed samples for only 1 hour at 500, 800, and 1000°C; his findings are important to analyze the possible outcomes of this study. In this study, specimens will be exposed to high temperatures and moisture conditions that will likely generate a stronger reaction in both DEF and ASR.

## 2.1.2 Calcium Sulfoaluminate Cements (CSA)

#### 2.1.2.1 Background and history

Since the sixties, CSAs have been used as low CO<sub>2</sub> generating alternative to Portland cements cementitious materials. Alexander Klein patented CSA cements as an expansive or shrinkage compensating addition to cementitious binders (Winnefeld, 2010). The active compound in the cement is calcium sulfoaluminate also known as "Klein's compound". The hydration of CSA cement is based on the fast formation of ettringite needles within the cement paste. The fast formation of ettringite generates a characteristic of rapid strength gain. In the 1970s, CSA's rapid setting and hardening feature allowed its utilization to be based on concrete repairs.

## 2.1.2.2 Cement chemistry and hydration of CSA

CSAs are produced by burning CSA clinker from limestone, bauxite, and calcium sulfate at about 1250°C which proceeds to blending clinker with 15 to 25 percent of gypsum and anhydrite. The CSA firing temperature for production is nearly 200°C lower comparing to OPC.

Ettringite is the principal hydration product of CSA. Ettringite precipitates with amorphous Al(OH)<sub>3</sub> until the calcium sulfate is consumed after a hydration cycle of one or two days (Winnefeld, 2010). Following the hydration cycle, monosulfate formation is generated.

According to Zhang et al, (2018), the main hydration phase for CSA consists in the presence of ye'elimite (C<sub>4</sub>A<sub>3</sub>S), and minor amounts of belite (C<sub>2</sub>S), anhydrite (CS) and gehlenite (C<sub>2</sub>AS). The first reaction of hydration is y'elimite involved with anhydrite

than then rises into ettringite and aluminum hydroxide. This reaction delivers properties such as low alkalinity and sulfate resistance.

Table 2. Comparison between OPC and CSA cements (Winnefield, 2010)

	OPC	CSA
Main phases	$C_3S$ , $C_2S$ , $C_3A$ , $C_4AF$	C₄A₃s (ye'elimite)
Raw materials	Limestone & clay	Limestone, bauxite & anhydrite
Burning temperature	1450°C	1250°C
Grindability	Medium	Easy
W/CM total hydration	0.4	0.6
Main hydration products	C-S-H phases, portlandite, ettringite, AFm-phases*	Ettringite, AFm-phases*, amorphous AI(OH) <sub>3</sub>

<sup>\*</sup>Afm phases is alumina, ferric oxide, monosulfate

#### 2.1.2.3 Application of CSA concrete

CSA cement is an innovative option in the construction industry that could replace Portland cement. CSA cement's popularity has emerged for having beneficial rapid setting characteristics and is being used for tunneling and underground construction projects. Department of Transportations across the United States has successfully used CSA cement concrete to replace thousands of lane-miles of freeway pavement. Featuring a 20-minute set time and great durability characteristics, CSA cement concrete has been used for rehabilitating concrete runways at airports throughout the world. Furthermore, CSA cements is beneficial for prestressed concrete construction by minimizing prestressing losses associated with shrinkage when OPC is utilized. The utilization of CSA cement is advantageous in events where construction interruptions could significantly affect the functionality of a facility such as airports or high traffic highways.

#### 2.1.3 Blended system incorporating CSA or CAC with OPC

# 2.1.3.1 Background and history

OPC is the hydraulic binder, obtained from limestone and clay that has been used for decades in the construction industry due to its reliable and excellent performance. Although CSA and CAC share qualities for developing fast setting, gaining high early strength and having a high content of Aluminum Oxide content, both systems have completely different microscopic crystal structures. CAC is a refractory cement mostly utilized for concrete repairs. CSA is pure powdered cement clinker, characterized for having a high content of Calcium Sulfoaluminate Crystals and mainly used for rapid repairs and precast products (Zhang et al, 2018).

#### 2.1.3.2 Cement chemistry and hydration of blended systems

CSA cements contain large portions of ye'elemite (C<sub>4</sub>A<sub>3</sub>S), which is also found in OPC, as well as belite and calcium sulfates. CSA cements react to from C-S-H and ettringite, similar to portland cements (Kurtis, 2019). However, the ettringite formed in CSA cements is greater and arises slower than binders containing only OPC. CAC contain mostly CA and react to form hydrated calcium aluminate phases. Usually, when CAC is blended with OPC, a certain amount of calcium sulfate is added. There are some phases in the hydration process that CAC and CSA have in common with OPC. All reactions in common contribute to setting and strength development.

Trauchessec et al, (2013) completed a study where OPC and CSA binders were analyzed. The study compared compressive strength and hydration processes of blends 85-15, 70-30, and 40-60% of OPC and CSA cement. 60% CSA cement blend had an

incremented hardening speed due to quick ye'elimite hydration. Whereas, for lower blends of CSA cement 15 and 30% hardening occurred slower. The report showed that OPC reacts slower during the first days and ye'elimite hydration occurs in the presence of lime to then form ettringite (Trauchesseec et al, 2013).

# 2.1.3.3 Application of blended systems

CSA binders are designed to blend with portland cement to make mixes that need fast setting and rapid hardening. CSA-OPC binders are characterized for providing mixtures freeze resistance, impermeability, low pH, corrosion resistance, and micro expansion properties (Kurtis, 2019). CAC and CSA are known for having high-cost values comparing to OPC. However, when blending CAC and CSA with OPC those costs can be balanced. The application of blended systems can be beneficial and not only used for repairs. The hydration process for both, CAC, and CSA, slows down once OPC is included in the binder. Therefore, blended mixtures have the characteristics of all three types of cements. Blended types lean towards OPC for having good malleability and CAC and CSA for features of sulfate resistance and rapid setting and strength gain. Blended binders can improve OPC dimensional stability (Trauchessec, 2013).

#### 2.2 Alkali-Silica Reaction

#### 2.2.1 Historical background

Alkali-silica reaction is a chemical interaction between siliceous constituents found in concrete aggregates and hydroxyl ions. Hydroxyl ions are found within concrete mainly because of the concentration of sodium and potassium (Virmani, 2014). The reactive silica found in the aggregates of most concrete mixes produces a reaction called

alkali-silica gel. Once this gel absorbs moisture, it expands which creates internal pressure in the concrete. The internal pressure can reach the point to exceed the tensile strength and crack the concrete. Therefore, potential ASR in concrete is studied, tested, and inspected to avoid cracking and weakening of the infrastructure.

ASR has visual indicators, such as map cracking, closed joints, and spalling. ASR has been widely known as one of the more prevalent deterioration mechanisms affecting concrete worldwide (Thomas, 2013). These problems were originally identified by Thomas Stanton in the State of California in the 1930s (Stanton, 1940).

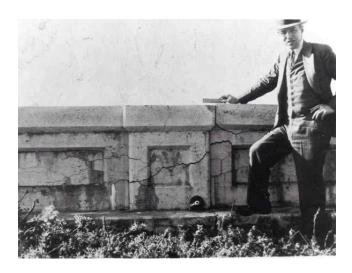


Figure 1. Thomas E. Stanton showing structure affected by ASR (Stanton, 1940)

Stanton discovered that the expansion of concrete structures was directly subjective to the alkali content of each concrete mixture, the magnitude of the reactive silica in the aggregates, and the exposure to different temperatures and moisture. Shortly after Thomas' findings were distributed around the construction industry the diagnosis of ASR turned out to be more common. This discovery also led to the initiation of studies

and deep investigations sponsored by agencies such as the Army Corps of Engineers, Bureau of Public Roads, Portland Cement Association, and others (Thomas, 2013).

## 2.2.2 Mechanisms and essential components

To have ASR damaging reaction occur, there are certain requirements that need to be present. Firstly, there must be a sufficient quantity of reactive silica within the aggregates. Secondly, there must be a sufficient concentration of alkali which is mainly characterized to be generated from portland cements, however, may also come from other constituents (i.e., SCMs, admixtures, and other powder-based binders). Lastly, plenty of moisture must be present in the environment where the structure is located (Thomas, 2013). Figure 2 displays the three requirements for concrete to subjected to ASR in the field as discussed by Thomas, et al. (2013).

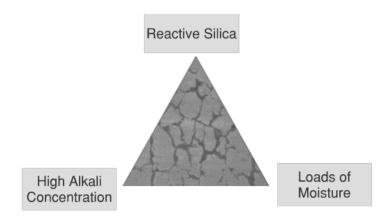


Figure 2. ASR components (Thomas, et al. 2013)

Although ASR has been identified to be affecting many of the world's concrete structures for over 75 years, its mechanisms to spot haven't yet been fully developed or proved. Its mechanisms start with the formation of alkali-silica gel which is mainly

composed of sodium, potassium, hydroxyl, and some amounts of calcium (Sims, 2016). Therefore, this gel absorbs water from the cement past which it surrounds. Once the gel absorbs a certain amount of water from the surrounding cement paste, the structure expands. The expansion of this water and gel absorption causes internal stress in the structure which ultimately leads to cracking (Figure 3).

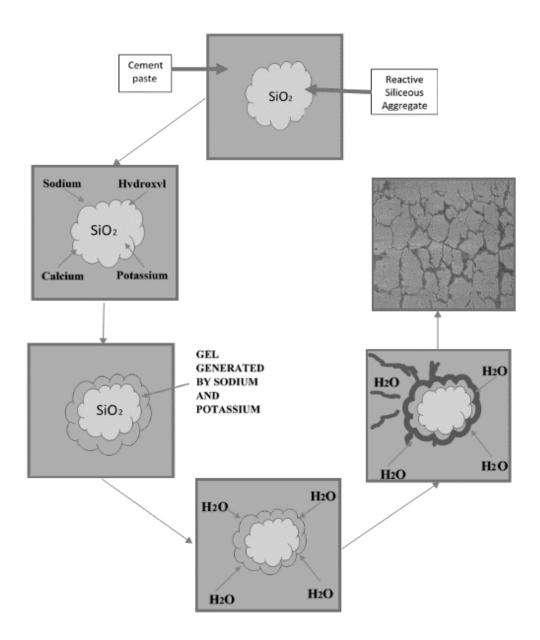


Figure 3. ASR Mechanism.

As previously mentioned, ASR is characterized for generating cracks in concrete structures. Although with time, many types of cracks can be seen in concrete constructions due to their normal deterioration. The cracks of ASR typically lean more to look like map shapes. In addition to producing cracks, ASR also generates expansion which can be restrained in one or more directions (Thomas, 2011).

Even though the signs of deterioration in concrete generated by the ASR manifest 5 to 10 years after construction, they can usually be identified during a routine site inspection which is performed regularly in job sites (Fournier, 2010). Structures located in cold and dry weather, might not show any ASR symptoms until the construction reaches an age of 15 years or more. The influence of temperature and weather conditions for ASR to react becomes a concern for the Texas infrastructures as the temperatures tend to be high and the weather is very humid almost all year long. According to the ASR field identification book published by the Federal Highway Administration, some of the common visual symptoms of ASR can range from surface pop-outs, cracking, crushing of concrete, discoloration on surfaces, gel production, expansion causing deformation and displacement of structures (Diamond, 1997).

The major symptom of ASR is a map cracking pattern which surfaces on concrete structures that are unrestrained (Thomas, 2013). Added to the cracks, discoloration can also occur. As shown below in Figure 4 the cracks take place in a randomly oriented form. These cracks are distinctive and can be seen in a variety of structures such as in concrete pavement and concrete columns of bridges.



Figure 4. Concrete Structures Affected by ASR (Thomas, 2013)

## 2.2.3 Laboratory and field-testing methods

#### 2.2.3.1 Accelerated Mortar Bar Test (ASTM C1260)

This method was developed by Oberholster and Davies in 1986 at the National Building Research Institute in South Africa (FHA, 2003). It provides a tool to identify the potential cause of ASR due to the utilization of a determined type of aggregate. Initially, ASTM C1260 was dedicated to only test the aggregate reactivity, but it had been found to be a great method to assess the levels of efficacy of supplementary cementing materials (SCMs) to reduce ASR expansion. The ASTM C1260 is used to determine whether an aggregate is potentially reactive.

The test is composed by a process involving the casting of mortar bars containing either coarse or fine aggregates. The following step in this test is to demold the mortar bars after 24 hours and place them in water at room temperature. The temperature of the water must be raised to 80°C in the oven, and the mortar bars specimens are stored in this new temperature and condition for another 24 hours. The bars are then removed from the water and measured to keep track of any length changes. After this process, the specimens are submersed in a 1 normal NaOH solution at

80°C to be stored for 14 days. The specimens are stored toward the soaking of the solution while periodically length measurements are taken.

In addition, the ASTM C1260 manages a type of expansion criteria. This criterion defines whether the specimen is innocuous, potentially reactive, and reactive referred in Table 1. Although these criteria limits are mentioned in the standard test method, they are not a mandatory part of the standard.

Table 3. Mortar Bar Expansion Values.

Expansion %	Considered as:
< 0.10 %	Harmless
0.10 to 0.20 %	Potentially reactive
> 0.20 %	Reactive

Even though the ASTM C1260 is suitable for aggregate reactivity testing, it is not a reliable source to assess alkalinity in cements. The solution used to soak the samples in this test method has a very high alkaline content that can cover any effect caused by alkalinity in the cement. According to the Portland Cement Association (2011), it is often more reliable and more common for researchers to use the ASTM C1293, *Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction*, which is also known as the concrete prism test or CPT. Though, ASTM C1260 had been used as a guidance for the creation of the mixture design. This standard is used to determine whether an aggregate is potentially reactive.

#### 2.2.3.2 Concrete Prism Test (ASTM C1293)

The ASTM C1293 concrete prism test is one of the most suitable and reliable methods to evaluate potential responses that will eventually lead to ASR. The ASTM

C1293 provides researchers a strong similarity that connects to the performance of specimens in the field. Additionally, this test is used to determine the susceptibility of an aggregate in expansive alkali-silica reaction by providing a way to measure the length changes of concrete prisms.

ASTM C1293 concrete prism test is utilized as the main method to evaluate potential responses that will eventually lead to ASR. ASTM C1293 is a long-term test. The results begin to show up around a year later after casting. The standard is meant to show the level of deleteriousness that the different mixtures could reach, referred in Table 4. It is well-thought-out for the specimen to fail the CPT expansion test if it expands more than 0.040% in one year.

Table 4. CPT Expansion Values (ASTM C1293)

Specimen to be considered as:	CPT Expansion %
Non-reactive	< 0.040
Moderately reactive	0.040-0.120
Highly reactive	0.120-0.240
Very highly reactive	>0.240

#### 2.2.3.3 Concrete Blocks

The purpose of developing concrete exposure blocks is to have a field trial to offer a more realistic sized element to evaluate ASR effects on different types of binder systems. The concrete blocks are considered as a field trial to analyze the effects of ASR in respect with all environmental conditions. Fournier et al. (2000) determined that interpreting the reactivity of aggregates in laboratory conditions is faster to obtain results but not as reliable as actually exposing the specimen to outdoor conditions. Thomas et al

(2006) also investigated that a specimen could fail ASTM C1293 in two years of age, nonetheless, it could take 10 years for an exposure block to surpass the 0.04% limit.

# 2.2.4 ASR performance of RSCH's

CAC and CSA cements are characterized for having early strength development.

RSCHs have generated highlights due to their lower energy consumption and environmental impact than the traditional OPC. Properties such as fast strength gain are based on the rapid formation of ettringite.

Types and dosage of activators are essential for ASR expansion in alkali-activated binders. As Wei (2020) mentioned, the most used activators are sodium hydroxide (NaOH), sodium silicate, and potassium hydroxide. These activators play an important role in the alkalinity activation, they all generate different chemical compositions and reactions which result in different ASR behavior in RSHCs systems. Therefore, this report manages only sodium hydroxide as an activator.

# 2.3 Delayed Ettringite Formation (DEF)

#### 2.3.1 Historical background

Ettringite is the mineral name for calcium sulfoaluminate that is usually found in OPC concretes. According to the Portland Cement Association, delayed ettringite formation (DEF) is referred to be a potential deleterious reformation of ettringite in moist concrete, mortar, or paste after the destruction of primary ettringite by high temperature. Ettringite stands as the mineral name for calcium sulfoaluminate (PCA, 2001). Ettringite that begins forming at early stages is known as "primary ettringite". The formation at early stages is part of an important component of Portland cement systems. Following,

ettringite is formed by the dissolution, and recrystallization in cracks is referred to as "secondary ettringite" and does not harm the performance of concrete (PCA, 2001).

DEF can affect large-scale elements of precast elements. The secondary ettringite formation is the result of the continuous dissolution of ettringite in fine cracks and cavities (Thomas et al, 2008). DEF is perceived as a complex phenomenon that never affects the entire structure (Pavoine, 2012). This structural deficiency has been seen around the world for many years, and it has been noted to be related to the exposure of the elements to high temperatures and the presence of water. Dayarathne (2013) explained DEF is defined as the result of a modification of the chemical reactions during hydration.



Figure 5. Concrete Structures Affected by DEF. (Thomas et al, 2008)

### 2.3.2 Mechanisms and essential components.

DEF is a heat-induced internal sulfate attack. This deficiency occurs when concrete is exposed to high temperatures at early stages such as curing. This internal

sulfate attack directly affects durability and strength of the concrete in a similar way to Alkali-Silica Reaction. Dayarathne (2013) also explained, besides the existing sulfate within the aggregate, there must be two important conditions for DEF to happen. The first condition is that the specimen's internal temperature must exceed 158°F / 70°C. The second condition is that there must be sufficient moisture available in the environment where the concrete exists. DEF is often connected with ASR; therefore, the use of potentially reactive aggregates can also exacerbate DEF.

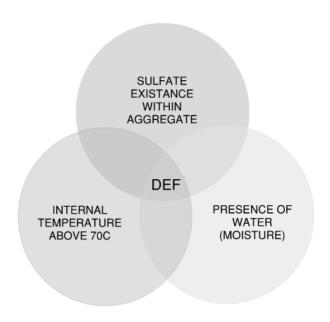


Figure 6. DEF Components

The absence of any of the conditions depicted in Figure 6 prohibits the formation of late ettringite. Rising temperatures can also alter the conditions of the equilibrium of the already existing sulfate within the aggregate. The sulfate content and sulfate/alumina ratio of the cements are contributing factors for DEF. In addition, for DEF to develop, temperature alterations must happen within the first 12 to 24 hours after mixing. DEF will not occur if the temperature of the specimen stays high for numerous days.

Figure 7 depicts the mechanism DEF uses to develop. Sulfate and alumina get confined rapidly leading them to form inner C-S-H sulfate when exposed to high temperatures. Sulfate and alumina slowly get released over time allowing pores to generate the formation of ettringite at later ages.

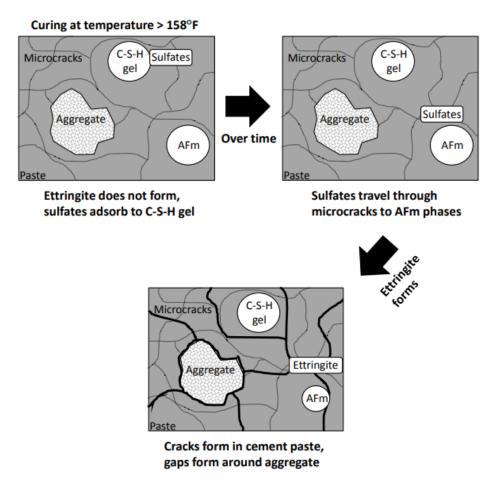


Figure 7. DEF Mechanism (Kreitman, 2011)

## 2.3.3 Testing methods

Presently, there are no standard ASTM or AASHTO test methods to obtain the possible effects of DEF in cementitious materials in a laboratory setting. The contributions of DEF are difficult to separate from the effects of distinct mechanisms

such as ASR. The Kelham could be addressed to accelerate how DEF could affect the cementitious material.

This project applied the reasoning from Kelham (1996) to cover molds with a water-soaked cloth to ensure 100% RH during curing. Samples were maintained at 23°C then subjected to a heat curing cycle. The curing regime according to the Kelham method reached 95°F. The Kelham Method differs from the Fu Method in the way that specimens are cured.

Fu developed a study in 1997 to define whether aggregates could cause reactivity generating expansion in specimens. Fu's study was based on thermal cycling to generate reactivity. His study was later referred as the Fu Method which was helpful for Kelham to acquire more knowledge in ettringite formation. Fu Method utilizes a curing method of oven drying the specimens, whereas, the Kelham Method only dries the specimens in a moist environment. The Kelham method is more reliable to expand the knowledge on DEF due to the management of the moisture conditions that can assimilate the most the Texas environment. In the cement and concrete research "Expansion of Portland cement mortar due to internal sulfate attack", Fu indicated that ettringite formation can be found in cement paste exposed to temperatures higher than 65°C when its drying, nonetheless the cement paste starts decomposing when drying occurs at 93°C.

#### 2.3.4 DEF performance of RSHC's

Currently, there are no studies that can be a tool of understanding how DEF reacts on CAC and CSA cements. RSHC's are known for reaching high temperatures at early stages of curing. Pavoine et al (2012) exposed the application of a method that can

accelerate the impact of DEF in cementitious materials that heat at temperatures over 65°C during curing. Pavoine et al (2012) took into count the sulphate content and the alkali content of the cements and concluded that the risk of DEF can be limited by reducing the amount of sulphate and alkali content.

#### 3. METHODOLOGY

### 3.1 Experimental Program

Laboratory and field investigations are carried out to investigate the susceptibility of CSA and CAC concrete to ASR. A combination of previous studies is to be presented as methodology for the development of this study. Preliminary and constant testing in the laboratory provided the tools to develop means and methods for a better understanding of local materials and their reaction with ASR and DEF.

Although, the size, mixture and exposure condition interpreted in the concrete prism method (ASTM C1293) and concrete exposure blocks are the most similar to real infrastructure, an extended amount of time is needed for this test to have complete results. According to Huang, who used and proved that concrete prism test method (CPT) to test the ASR expansion in alkali-activated systems was suitable, it took one year for OPC and two years for mitigation methods to obtain results regarding development and practicability. Therefore, this study also includes three series of mortar bar methods. The Mortar-bar method will be utilized to accelerate the hydration and strength development of the specimens.

### 3.2 Materials and mixture proportions

Concrete is a material formed by a mix of coarse aggregate, fine aggregate, cement, and water (West, 1996). Normally, the proportions of concrete composition are about 80% aggregate, 13% cement, and 7% water. Coarse aggregates are materials greater than 2.36 mm in diameter. Fine aggregates that are less than 2.36 mm are often defined as sand.

Cement is the material that binds together and strengthens concrete. Assessing the right aggregate can help the concrete to have a better long-term quality.

Stanton (1940) proved that the size of the reactive aggregate is an important factor that affects the magnitude of the alkali-silica reaction. An important reason to be cautious when selecting the right type of aggregate is that the aggregate in each mixture is a major factor that supplies reactive silica (Virmani, 2014). Aggregates can be obtained from different locations and have the unique capacity to be differentiated by their composition, size, and chemical properties.

The most effective way to avoid obtaining false-positive results when exposing specimens to ASR and DEF testing is to use materials that will not react. Consequently, the materials chosen for this report are listed in Table 5. All three aggregates used in this project were obtained from Central and South Texas producers. Throughout the entire work, these aggregates will be referred to as Jobe, Limestone, and El Indio. Although Limestone and El Indio are coarse aggregates, they were crushed and respectively sieved in the laboratory.

Table 5. Aggregates: Types, Categories and Origins

Name of aggregate	me of aggregate Type		Origin		
Jobe	Fine aggregate	Reactive	El Paso, TX		
Limestone	Coarse aggregate	Non-reactive	San Antonio, TX		
El Indio	Coarse aggregate	Moderately reactive	Eagle Pass, TX		

Composing 13% of the cementitious mixture, cement is an additional aspect of influence for ASR and DEF to develop. A variety of cements are manipulated in this research study to get a broader understanding of ASR and DEF affecting RSHC's. The cements listed in Table 6 are utilized for this report.

The materials that are exclusively manufactured to contain only one type of cement, which in this case is either portland, calcium aluminate or calcium sulfoaluminate cement; are referenced as pure cements. Straight cements such as Type I (OPC1), Type I/II (OPC2), and Type III (OPC3) are known for having good workability turning them into the most used type of cements in the construction industry. Per manufacturers' indications, three pure rapid setting hardening cements were included in the study such as CAC1, CSA1 and CSA2. These cements are standalone cements and do not require blending or additives to achieve superior performance.

Even though, the composition of proprietary blended cements is not strongly clarified by the manufacturers. It is known that proprietary blended cements are already manufactured containing a certain amount of OPC clinker. Furthermore, laboratory blended cements are used to analyze the influence of Type I/II (OPC2) in the performance of rapid setting cements such as CAC and CSA1. All laboratory blended systems were proportioned to contain a 25 to 75% OPC and RSHC, respectively. For reference the blend type, cement IDs, and cement types can be appreciated in Table 6.

Table 6. Cements: Blend Types, Tags and Types

Blend Type	Cement ID	<b>Cement Category</b>	Description			
	OPC1	Portland	Normal setting portland cement with high alkali			
	OPC2	Portland	Normal setting portland cement, moderate C <sub>3</sub> A, and low alkali			
Straight	OPC3	Portland	Rapid Setting Portland Cement used as control for RSHC			
Cements	CAC1	Calcium Aluminate	Rapid Setting Cement based on Calcium Aluminate			
	CSA1	Calcium Sulfoaluminate	Rapid Setting Cement based on Calcium SulfoAluminate with low belite system			
	CSA2	Calcium Sulfoaluminate	Rapid Setting Cement based on Calcium SulfoAluminate with high belite system.			
	CAC-B1	Calcium Aluminate	Proprietary Rapid Setting Cement based on Calcium Aluminate and OPC			
	CAC-B2	Calcium Aluminate	Proprietary PreBlend of CAC with Class C Fly Ash			
Proprietary Blended	CSA2-B1	Calcium Sulfoaluminate	Proprietary PreBlend of CSA with pozzolan and mineral admixtures			
Cements	CSA2-B2	Calcium Sulfoaluminate	Proprietary PreBlend of CSA with pozzolan and mineral admixtures			
	PCSA1	Calcium Sulfoaluminate	Proprietary PreBlend CSA			
	PCSA2	Calcium Sulfoaluminate	Proprietary PreBlend CSA but with different chemistry and/or fineness			
Laboratory Blended	CAC-OPC2	Calcium Aluminate with Portland	Ternal NT 204 is a pure blend of CAC at a ratio of 2.2:1. This systems is intended to be blended with local OPC.			
Cements	CSA-OPC2	Calcium Sulfoaluminate with Portland	CSA is a pure low belite CSA system. This binder is can be used on its own or in combination with OPC.			

Granting that the specific constituents added on these proprietary blended cements are not available from the manufacturers, the chemical composition is detailed in Table 7. Cement chemistry is thought to be a major contributing factor for reactions related to ASR and DEF. The amount of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), calcium oxide (CaO), sulfate (SO<sub>3</sub>), and alkalis (Na<sub>2</sub>Oe) are predictable to be inducing factors for ASR and DEF effects.

Table 7. Cements: Chemical Properties

Blend Type	ID	SiO2	Al2O3	Fe2O3	CaO	MgO	SO3	Na2O	K2O	Na2Oe	P2O5	Cl	TiO2	MnO	ZnO	Cr2O3	LOI	CO2
	OPC1	19.60	5.19	2.06	64.01	1.12	3.86	0.12	0.91	0.72	0.13	0.01	0.24	0.03	0.01	0.01	3.80	2.49
	OPC2	21.06	4.02	3.19	63.91	1.08	2.89	0.14	0.61	0.53	0.11	0.01	0.18	0.03	0.05	0.01	2.29	1.52
Pure	OPC3	19.67	5.34	1.76	63.41	0.99	5.27	0.10	0.44	0.39	0.29	0.01	0.23	0.03	0.01	0.01	4.06	1.19
Cements	CAC	4.34	38.65	15.09	38.37	0.39	0.16	0.05	0.14	0.14	0.12	0.00	1.82	0.11	0.02	0.11	1.55	0.64
	CSA1	9.07	21.61	2.26	45.26	0.94	20.26	0.07	0.30	0.27	0.07	0.01	0.76	0.07	0.01	0.02	1.05	0.27
	CSA2	20.56	16.14	1.35	45.31	1.23	14.73	0.77	0.72	1.24	0.16	0.02	0.76	0.01	0.02	0.02	4.74	1.81
	CAC-B1	13.46	12.23	2.67	56.65	2.86	9.90	0.20	0.79	0.72	0.11	0.01	0.60	0.14	0.07	0.04	1.21	0.54
Duon	CAC-B2	12.71	32.94	12.95	35.09	1.79	0.84	0.50	0.24	0.65	0.30	0.01	1.70	0.09	0.02	0.09	1.23	0.36
Prop	CSA2-B1	13.63	15.82	0.75	51.28	1.14	16.62	0.29	0.62	0.69	0.15	0.02	0.72	0.01	0.02	0.02	3.06	1.28
Blended Cements	CSA2-B2	14.72	14.37	1.22	53.85	1.23	14.40	0.10	0.59	0.49	0.15	0.02	0.65	0.04	0.01	0.02	3.39	1.76
Cements	PCSA1	17.38	11.06	2.98	55.82	1.25	10.68	0.43	0.52	0.77	0.12	0.01	0.58	0.07	0.01	0.01	2.26	1.25
	PCSA2	20.14	15.73	3.52	43.90	1.55	12.88	0.59	0.52	0.93	0.23	0.01	0.75	0.06	0.02	0.03	1.95	0.82
Lab	CAC-OPC2	16.53	10.79	2.71	58.07	0.89	7.43	0.14	0.50	0.47	0.10	0.01	0.52	0.02	0.04	0.02	2.19	1.23
Blends	CSA-OPC2	18.06	8.42	2.96	59.25	1.04	7.23	0.12	0.53	0.47	0.10	0.01	0.33	0.04	0.04	0.01	1.98	1.21
N/A	CAC1	2.93	31.12	1.25	40.57	0.33	21.08	0.14	0.19	0.27	0.07	0.00	1.54	0.01	0.01	0.04	1.90	0.37

Admixtures are adjusted and added accordingly to the workability needs while mixing.

The admixtures of utilization are listed and described in Table 8.

Table 8. Admixtures: Description and Potential Outcomes

Admixture	Description	Potential outcome/effects				
Viscocrete 4100	High range water reductor	Achieve maximum water reduction				
Sika NC	Chloride-free set accelerator	Accelerate set times and increase early strength gain. Recommended 10-45 fl. Oz. per 100 lbs of cement.				
GCX-500	CAC based accelerator	Accerelate set times. Recommended by CAC cement supplier.				
Citric acid powder	Organic retarder	Doses between 0.2% and 0.4% for moderate retardation. Potential to lead to a slower growth of silicate crystal.				
Sodium Hydroxide (NaOH)	50% solution	Used for non-alkali-reactive aggregates.				

## 3.3 Alkali-Silica Reactivity Experimental Methods

## 3.3.1 Concrete exposure blocks

At the time of analyzing ASR, the only benchmarking of a laboratory performance test is against real concrete structures if available. However, there can be surrogate for ASR studies, which are large concrete blocks exposed outdoors and exposed to natural weathering conditions Researchers such as, Thomas (2006) have also employed the use of large concrete exposure blocks subjected to natural conditions to determine the vulnerability of an aggregate or combination of aggregate in expansions due to ASR (Thomas, 2006).

An exposure site within the facilities of Texas State University in San Marcos, Texas was organized in July of 2020. A total of 12 concrete mixtures were prepared in August of 2020, shown in Figure 8. These blocks are monitored periodically to obtain findings on possible cracking generated by ASR. The monitoring of these exposure blocks includes visual inspections, length change measurements and, temperature and relative humidity variations. Although the blocks currently range between six and eight months of age, only six-month data are taken into count for writing compilation and analyses.



Figure 8. Exposure Concrete Block Site at Texas State University - February 15, 2021

## 3.3.1.1 Materials and mixture proportions

Previous studies performed in the laboratory, allowed the development of all mixture designs. Being the most common type of cement used in the construction industry, Type I (OPC1) served as the baseline material for comparison against CSA and CAC cements. OPC1 containing an alkali content of 0.72 Na<sub>2</sub>Oeq was used. The alkali content in CSA and CAC cements varied between 0.14 and 1.24 as described in Table 9.

This binder of concrete blocks managed a low water/cement ratio of 0.35, a total cementitious content of 752 per cubic yard and no additional alkalis added into the mixtures. Furthermore, the series is described as undoped throughout the project due to the lack of additional Sodium Hydroxide (NaOH). In addition to the exposure blocks, this mixture matrix included (4) prisms for series 1 CPT test and (2) cubes for strength purposes.

Table 9. Concrete Exposure Blocks: Mixture Design

Mix Tag	Primary Cement*	Secondary Cement* Type I/II	Rock*	Sand*	Water*	NaOH*
OPC1	752.00	-	1844.16	1152.60	263.20	-
OPC2	1	-	1	-	-	-
OPC3	1	-	1	-	-	-
CAC1	752.00	-	1844.16	1152.60	263.20	-
CSA1	752.00	-	1844.16	1152.60	263.20	-
CSA2	658.00	-	1913.57	1195.98	250.04	-
CAC-B1	752.00	-	1844.16	1152.60	263.20	1
CAC-B2	752.00	-	1844.16	1152.60	263.20	-
CSA2-B1	752.00	-	1844.16	1152.60	263.20	ı
CSA2-B2	752.00	-	1844.16	1152.60	263.20	1
PCSA1	752.00	-	1844.16	1152.60	263.20	-
PCSA2	752.00	-	1844.16	1152.60	263.20	ı
CAC-OPC2-B1	188.00	564.00	1844.16	1152.60	263.20	-
CSA-OPC2-B2	188.00	564.00	1844.16	1152.60	263.20	-
CAC-OPC2-B2	-	-	-	-	-	-
CSA-OPC2-B2	-	-	-	-	-	-

<sup>\*</sup>Weight in Pounds per Cubic Yard

## 3.3.1.2 Timeline and procedure

The exposure blocks series is the most realistic series of binders throughout the project. The 16"x16" concrete exposure blocks are the biggest size of specimen available for ASR testing. These blocks are the most comparable in size to concrete structures that are normally used in heavy civil projects.

Concrete blocks were kept at outdoor exposure with the aim of experiencing the effects of all temperatures and humidity levels in San Marcos, TX. The standardized test ASTM C1293 was applied toward mixing processes and to determine ASR effects on CAC, CSA, and blended binders cast as concrete exposure blocks.

The timeline started at day 0 which is the day the concrete was poured. To accelerate curing, the temperature and relative humidity must reach their highest point. Therefore, the specimen was covered with a slightly wetted burlap and a plastic layer as soon as pouring was completed. Each block maintained a curing regime of 7 days. On day 7, the specimens were demolded.



Figure 9. (1) Exterior View of 16 x 16 x 16 in Wooden Mold (2) Side View of Wooden Mold (3) Interior View of Wooden Mold.

According to ASTM C1293 for ASR findings, the timeline for completion is typically 24 months. Expansion and drying shrinkage of the specimens was captured at specific ages. The specimens were measured in the length comparator at age of 7, 28, and 56 days, as well as 3, 6, 9, 12, 18 and 24 months.

Each block specimen contained a total of 4 stainless steel pins on each face, for a total of 20 for measuring purposes. The measurements were taken monthly for about six to eight months. Each measurement includes twelve expansion measurements that were averaged at each exposure time. The gauge-like length comparator was adjusted according to the side of the block to be measured.

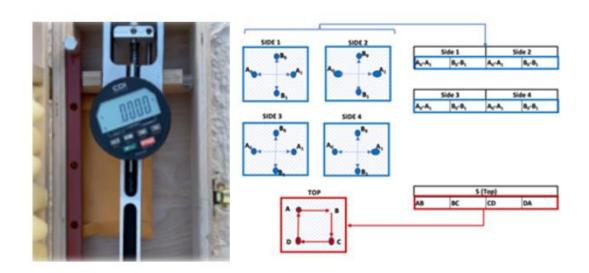


Figure 10. (1) Gauge Measurement Device (2) Pin Measurements Diagram

As clarified in method ASTM C1293, the ideal exposure time to develop measurements range between 68-77°F. Therefore, a great tool to obtain accurate temperatures and measurements for the expansion blocks was a concrete sensor. The concrete sensor from Hilti was added precisely before pouring, as shown in Figure 11. It

was attached to one of the pins inside the concrete block to measure relative humidity and temperature. This sensor was activated with light and connected via Bluetooth to a mobile phone. The temperature and humidity data obtained from each sensor within each mixture was directly extracted from the mobile application to ensure constant values for the time of measurements. Due to the shortage of equipment availability, only nine out of twelve mixtures contained sensors.

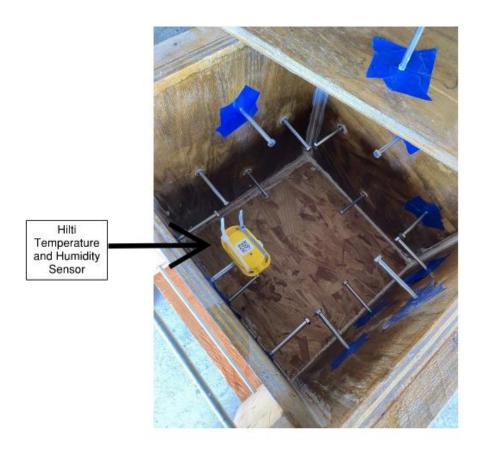


Figure 11. Interior View of 16 x 16 x 16 in Wooden Mold with Hilti Temperature and Humidity Sensor Attached

Ideally, the completion date for the study is 24 months after the casting date. All concrete exposure blocks in this report were analyzed only until 6 months of age. Figure 16 shows the finished results of each concrete block sample. Each block was properly labeled marking 5 sides with 4 pins each to allow tracking of measurements. The vertical sides of steel pins were labeled as to A0-A1 and the horizontal steel to as B0-B1.





Figure 12. Completed Concrete Exposure Block – CSA2

#### 3.3.2 Concrete Prism Test (ASTM C1293)

## 3.3.2.1 Materials and mixture proportions

Three series built accordingly to method ASTM C1293 and a modified version of it. As referred to in ASTM C1293, NaOH is a reagent. The value of 1.25% Na2O equivalent by mass of cement was chosen to accelerate the process of expansion rather than to reproduce field conditions.

The development of three series of binders will be the main basis of results for CPT testing. These mixtures were prepared using both changing waters to cementitious material ratio and constant water to cementitious material ratio.

Series 1: Series 1 was a modified version of the standard Concrete Prism Method (ASTM C1293). This binder managed a low water/cement ratio of 0.35, total cementitious content of 752 pounds per cubic yard, and no additional alkalis added into the mixtures. Furthermore, the series stated as undoped due to the lack of additional Sodium Hydroxide (NaOH). The mixture design per cubic yard can be appreciated in Table 8. Series 1 mixture design fell inside the quantities described in Table 10 which was prepared for the concrete exposure blocks. Series 1 contained a total of 12 mixtures casted as (4) 3"x3"x11.25" prisms, and (2) 4"x8" cylinders for strength tests.

Table 10. CPT: Series 1 Undoped Mixture Design

Mix Tag	Primary Cement*	Secondary Cement* Type I/II	Rock*	Sand*	Water*	NaOH*
OPC1	752.00	-	1844.16	1152.60	263.20	ı
OPC2	-	-	-	-	-	ı
OPC3	-	-	-	-	-	ı
CAC1	752.00	-	1844.16	1152.60	263.20	-
CSA1	752.00	-	1844.16	1152.60	263.20	-
CSA2	658.00	-	1913.57	1195.98	250.04	-
CAC-B1	752.00	-	1844.16	1152.60	263.20	-
CAC-B2	752.00	-	1844.16	1152.60	263.20	ı
CSA2-B1	752.00	-	1844.16	1152.60	263.20	ı
CSA2-B2	752.00	-	1844.16	1152.60	263.20	-
PCSA1	752.00	-	1844.16	1152.60	263.20	ı
PCSA2	752.00	-	1844.16	1152.60	263.20	ı
CAC-OPC2-B1	188.00	564.00	1844.16	1152.60	263.20	ı
CSA-OPC2-B2	188.00	564.00	1844.16	1152.60	263.20	1
CAC-OPC2-B2	-	-	-	_	-	i
CSA-OPC2-B2	-	-	-	-	-	-

<sup>\*</sup>Weight in Pounds per Cubic Yard

Series 2: Considered also as a modified version of the Concrete Prism Method (ASTM C1293). It used a low water/cement ratio of 0.35 and a total cementitious content of 752 per cubic yard. The addition of Sodium Hydroxide allows the aggregate to have a stronger reaction to the potential alkali-silica effects (Virmani, 2014). Therefore, Series 2 contained additional alkalis that compose an equivalent alkali content of 1.25% with the addition of a 50/50 NaOH solution. The mixture design per cubic yard can be seen in Table 11. This doped binder will also contain a total of 12 mixtures casted as, (4) 3"x3"x11.25" Prisms and (2) 4"x8" cylinders for strength tests.

Table 11. CPT: Series 2 Doped Mixture Design

Mix Tag	Primary Cement*	Secondary Cement* Type I/II	Rock*	Sand*	Water*	NaOH*
OPC1	752.00	-	1842.87	1151.79	260.61	5.18281
CAC1	752.00	-	1841.47	1150.92	257.82	10.7576
CSA1	752.00	-	1841.78	1151.11	258.44	9.5255
CSA2	752.00	-	1804.88	1128.05	282.08	7.3546
CAC-B1	752.00	-	1842.87	1151.79	260.61	5.1791
CAC-B2	752.00	-	1878.96	1174.35	237.74	5.79921
CSA2-B1	752.00	-	1806.55	1129.10	283.05	5.42709
CSA2-B2	752.00	-	1806.07	1128.79	282.07	7.37788
PCSA1	752.00	-	1842.99	1151.87	260.86	4.67907
PCSA2	752.00	-	1843.38	1152.11	261.64	3.11968
CAC-OPC2-B1	188.00	564.00	1842.26	1151.41	259.40	7.59706
CSA-OPC2-B2	188.00	564.00	1842.26	1151.42	259.41	7.5893

\*Weight in Pounds per Cubic Yard

Series 3: This series was based on the standard Concrete Prism Method (ASTM C1293). Using a water/cement ratio of 0.42 and a total cementitious content of 705 per cubic yard. Series 3 was also doped meaning that it contained a total equivalent alkali content of 1.25% with the addition of a 50/50 NaOH solution. The mixture design per cubic yard can be seen in Table 12. This doped binder also contained a total of 12 mixtures casted as, (3) 3"x3"x11.25" Prisms and (2) 4"x8" cylinders for strength tests.

Table 12. CPT: Series 3 Doped Mixture Design

Mix Tag	Primary Cement*	Secondary Cement* Type I/II	Rock*	Sand*	Water*	NaOH*
OPC1	704.98	ı	1814.23	1133.89	293.66	4.86
CAC1	704.98	ı	1812.92	1133.08	291.05	10.09
CSA1	704.98	-	1813.21	1133.26	291.63	8.93
CSA2	704.98	-	1813.72	1133.58	1133.58	6.89
CAC-B1	704.98	-	1814.23	1133.89	293.67	4.86
CSA2-B1	704.98	-	1814.17	1133.86	293.55	5.09
CSA2-B2	704.98	-	1813.71	1133.57	292.63	6.92
PCSA1	705.41	-	1813.85	1133.65	294.08	4.39
PCSA2	704.98	-	1814.71	1134.20	294.63	2.92
CAC-OPC2-B1	176.25	528.74	1813.66	1133.54	292.53	7.12
CSA-OPC2-B2	176.25	528.74	1813.66	1133.54	292.54	7.11
	*W	eight in Poun	ds per Cubi	c Yard		

## 3.3.2.2 Timeline and procedure.

Expansion and drying shrinkage of the specimens was captured at specific ages. The specimens were measured in the length comparator at ages of 7, 28, and 56 days, as well as 3, 6, 9, 12, 18, and 24 months. A gauge length comparator with a digital indicator was used to obtain precise measurements of length changes in the specimens. The specimens were placed in a container within the storage area. The container had a cloth covering its inner vertical surface and a plastic base to keep the specimens standing without having contact with the water to increase the humidity.



Figure 13. (1) Container Storing Prims ASTM C1293 (2) Measurement Device Following ASTM C1293 test method provided a means of detecting the potential of an aggregate intended for use in concrete for undergoing ASR resulting in potentially deleterious internal expansion.

#### 3.3.3 Accelerated Mortar Bar Test (ASTM C1260/C1567)

## 3.3.3.1 Materials and mixture proportions

The specimens used for the Accelerated Mortar Bar Test included (4)

1"x1"x11.25" prisms per mixture. Each series contain 16 different types of cement
varying between straight, blended proprietary, and lab blended cements. The
differentiation for the series is directly correlated to the type of aggregate used.

Aggregates such as Jobe, El Indio, and Limestone were used for this study. All three
series manage the same w/cm ratio, and the binder control is 100% for straight and
blended proprietary, whereas the lab blended cements maintain a control binder between

25% to 75% depending on the type of cement.

The procedure of this test started in the sieve analysis which determined the gradation to comply with ASTM C1260 / C1567. The specifications included sieve grades shown in Table 13.

Table 13. Sieving Grades for El Indio, Limestone and Jobe

Sieve #	mm			
No. 8	2.36			
No. 16	1.18			
No. 30	0.6			
No. 50	0.3			
No. 100	0.15			

The moment the aggregate is sieved, each grade was weighted accordingly to Table 14. The sand was sieved correspondingly to ASTM C1260.

Table 14. Fine Aggregate Grading

Fine Aggregate	Mass (%)	Mass (grams)
No. 8	10%	132
No. 16	25%	330
No. 30	25%	330
No. 50	25%	330
No. 100	15%	198
Sub-total	100%	1,320.00

The precise identical mixture design was carried among the three series that compose the ASR Mortar Bar Test in the project. The mixture design given in Table 15 used Jobe, Limestone and El Indio as fine aggregates.

Table 15. Mortar Bar Test ASTM C1260: Mixture Design

M(2 /ID	<b>Base Cement</b>	Type I/II		Fine A	Aggrega	te (g)		Water
Mix Tag	<b>(g)</b>	(g)	No.8	No.16	No.30	No.50	No.100	(g)
OPC1	586.70	ı	132.00	330.00	330.00	330.00	198.00	299.25
OPC2	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
OPC3	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC1	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA1	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2	586.70	ı	132.00	330.00	330.00	330.00	198.00	299.25
CAC-B1	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC-B2	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2-B1	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2-B2	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
PCSA1	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
PCSA2	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC-OPC2-B1	440.03	146.68	132.00	330.00	330.00	330.00	198.00	299.25
CSA-OPC2-B2	440.03	146.68	132.00	330.00	330.00	330.00	198.00	299.25
CAC-OPC2-B2	146.68	440.03	132.00	330.00	330.00	330.00	198.00	299.25
CSA-OPC2-B2	146.68	440.03	132.00	330.00	330.00	330.00	198.00	299.25

The preparation of the 1N sodium hydroxide solution was based off the use of Sodium Hydroxide (NaOH) pellets. The sodium hydroxide solution was prepared in accordance with ASTM C1260 / C1567. The 1N solution was prepared to correspond to every single mixture.

## 3.3.3.2 Timeline and procedure

The mixing of mortar procedure was made according to ASTM C305. After molds were filled with mortar paste, each mold was placed to cure in regular room temperature. The molds were covered by a slightly wetted burlap and a layer of plastic to maintain a high relative humidity. The specimens were cured for 24 hours. At 24 hours, demolding took place and initial measurement were taken.

The specimens were then placed in room temperature sealed water bath containers and set in the oven at 80°C for 24 hours. Simultaneously, sealed containers holding 1M NaOH solution were placed in the same oven at 80°C. Specimens made with unique cementitious materials and aggregates were stored in separated containers, as shown in Figure 14. Once the 24 hours are completed, the containers filled with water carrying the specimens are removed from the oven, dried, and carefully measured. The process of drying and measuring had to be completed within 15 seconds of removing the specimen for the water to solution. This measurement is attributed as day zero reading.

After recording day zero readings, the specimens were rapidly transferred to the NaOH solution containers that had been placed at the same time as the water containers. The water containers were then emptied and discarded. The NaOH solution containers storing the specimens were kept in the oven at 80°C and extracted for measurements on days 3, 5, 7, 10, 14, 21 and 28. On day 28, the specimens and containers with solutions were properly disposed after the final measurement.



Figure 14. Mortar Bars Submerged in NaOH Solution and Measurement Apparatus

## 3.4 Delayed Ettringite Formation experimental methods

## 3.4.1 Accelerated high temperature curing (Kelham Test Method)

The testing for susceptibility of concrete to DEF started with casting test prisms and curing them at high-temperature regimes to simulate high internal temperatures in concrete during curing. Consequently, test prisms were measured over time as ettringite continues to form in the concrete pores. Even though there are no standardized methods to determine DEF, Kelham (1996) developed a high temperature curing regime to activate the release of ettringite in cement concrete.

The Kelham Method was chosen as a basis for this study. Two series of binders were prepared to investigate in-depth the effect of temperature in the generation of DEF in rapid setting cements. Both set of binders managed the same mixture proportions with the difference of their thermal cycling for curing. The curing cycle utilized by Kelham was based on a peak temperature of 95°C (203°F) for 12 hours. Series 2 referred to as the Modified Method throughout the project. The Modified method maintained a peak temperature of 65°C (149°F) as shown in Figure 15 and 16 respectively.

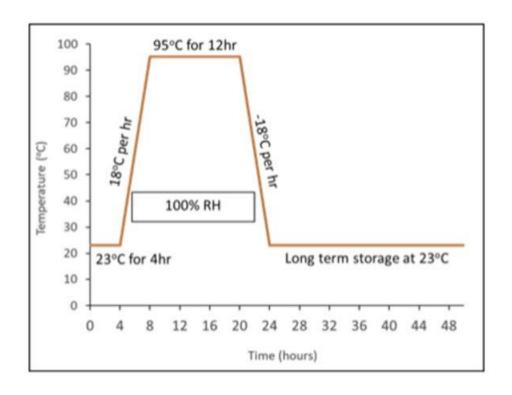


Figure 15. Series 1: Kelham Method for DEF Curing

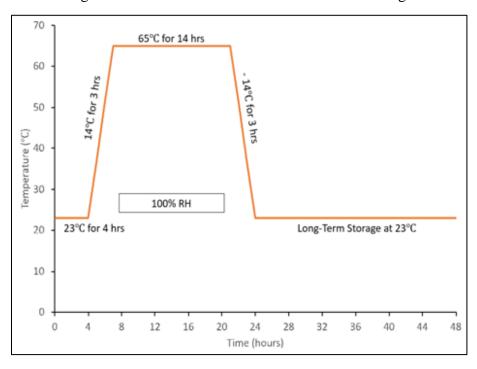


Figure 16. Series 2: Modified Method for DEF Curing

### 3.4.1.1 Materials and mixture proportions

Series 1 and 2 consisted of the exact same set of binders. Both series contained the same materials, and proportions with the only difference of the curing cycles. Each series was composed of 14 standard mortar mixtures which include (4) 1"x1"x11.25" prism specimens with a w/cm of 0.47. Both series were prepared with limestone which is a local non-reactive aggregate extracted from San Antonio, TX. Each mixture contained a cementitious ratio of 2.25 ad a w/cm ratio of 0.47.

Table 16. DEF Series 1 and 2: Mixture Design

		Base	Type I/II		Fine .	Aggregat	e (g)		Water
Mix Tag	Cement Type	Cement (g)	(g)	No.8	No.16	No.30	No.50	No.100	(g)
OPC1	OPC Type I	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
OPC2	OPC Type I/II	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
OPC3	OPC Type III	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC1	Fondu, CAC	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA1	CSA BUZZI	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2	CSA CTS	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC-B1	PP 5001	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC-B2	Quadra Set	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2-B1	CSA CTS	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CSA2-B2	CSA CTS	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
PCSA1	Fas Trac 300	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
PCSA2	Fas Trac 400	586.70	-	132.00	330.00	330.00	330.00	198.00	299.25
CAC-OPC2-B1	CAC Ternal 25%	440.03	146.68	132.00	330.00	330.00	330.00	198.00	299.25
CSA-OPC2-B2	CSA Buzzi 25%	440.03	146.68	132.00	330.00	330.00	330.00	198.00	299.25
CAC-OPC2-B2	CAC Ternal 75%	146.68	440.03	132.00	330.00	330.00	330.00	198.00	299.25
CSA-OPC2-B2	CSA Buzzi 75%	146.68	440.03	132.00	330.00	330.00	330.00	198.00	299.25

### 3.4.1.2 Timeline and procedure

The mixing procedure was performed according to ASTM C1260/C1567. After molds were filled with mortar paste, each mold was placed in a moist container. The moist container was prepared with a layer of water and plastic bases to avoid the contact of the molds with the water.

The curing regime started holding a 23°C temperature for 4 hours. It then proceeded to heating up at 20°C per hour to get to 95°C. The 95°C temperature was held for 12 hours to then proceed to cool at 20°C per hour. The temperature dropped to get back to 23°C exactly at 24 hours after the pour.

In contrast, the Modified Method raised the temperature by 16°C per hour to get to a maximum temperature of 65°C. The 65°C temperature was also held for 12 hours and proceeded to cool at 16°C per hour. Once the temperature lowered to 23°C, the specimens completed 24 hours of age. Proceeding the pouring and curing regime, the specimens were demolded, and measured. Both, the Kelham Method and The Modification Method managed a regimen to maintain specimens stored in a saturated solution of lime water at 23°C long-term. The lime water was prepared according to ASTM C31 Section 7.3 Lime water is a hydrated solution that contains Calcium Hydroxide (Ca(OH)<sub>2</sub>). The utilization of Calcium Hydroxide aimed to accelerate the release of ettringite in the specimens.

### 4. RESULTS AND DISCUSSION

### 4.1 Alkali silica reaction

# 4.1.1 Concrete exposure blocks

At the time of this writing, most of the specimens surpassed 6 months of age. However, this study limited the analysis to 6 months of age (183 days). Table 17 shows the average data obtained at an age of 183 days (6 months).

Table 17. Concrete Exposure Blocks: Average Expansion Days 28 through 183

Mixture	Day					
	28	56	84	121	152	183
OPC1	-0.0236	-0.0227	-0.0322	-0.0270	0.0111	0.0049
CAC1	0.0234	0.0254	0.0010	-0.0213	-0.0366	0.0041
CSA1	0.0109	0.0065	0.0113	0.0310	-0.0176	-0.0004
CSA2	0.0000	0.0233	0.0294	0.0210	0.0295	0.0219
CAC-B1	0.0035	-0.0167	0.0011	0.0620	0.0485	0.0309
CAC-B2	0.0255	0.0567	0.0726	-	0.0553	0.0365
CSA2-B1	0.0051	0.0171	0.0216	-	0.0163	0.0200
CSA2-B2	0.0447	0.0727	0.0790	-	0.0582	0.0542
PCSA1	-0.0031	0.0066	-0.0006	-0.0090	0.0349	0.0358
PCSA2	0.0260	0.0267	0.0320	0.0333	0.0645	0.0836
CAC-OPC2	0.0152	0.0252	0.0135	0.0059	0.0027	-0.0006
CSA-OPC2	0.0018	-0.0115	0.0202	-0.0199	-0.0266	0.0061
* Measurements given in percent						
- Indicate data skipped due to cold temperatures						

The results on expansion measured over 6 months for all 12 mixes are shown in Figure 17. The red dotted line expressed in the graphs throughout the analysis of ASTM C1293-18a, represents the limit of failure for the blocks with respect to the standard.

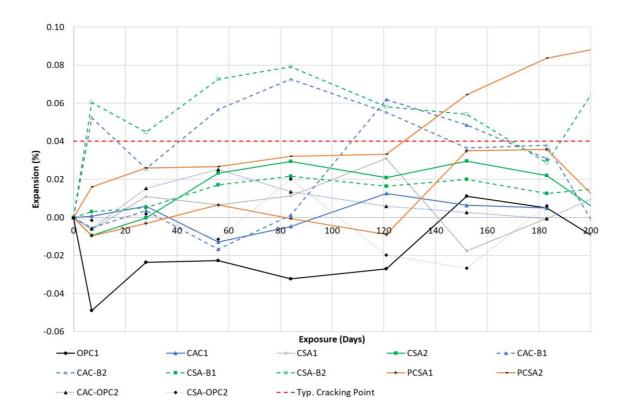


Figure 17. Overall Expansion Rates for CSA, CAC, and OPC in Exposure Blocks.

When considering which cementitious material is the least reactive to ASR, it was taken into consideration the division of the type of cement and blend. Figure 18 is the graphical representation of the CAC systems that did not exceed 0.04% to fail ASTM C1293.

CAC1 showed continued expansion throughout the 6 months of age. In this study, CAC1 cement had the lowest alkali content of 0.14. The constant and low expansion of the CAC1 mixture may perhaps be attributed to the low content of alkalis within the cement.

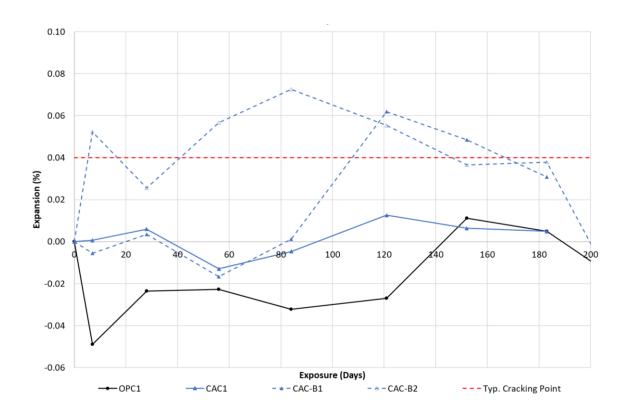


Figure 18. CAC System Expansion Rates

CAC1 exposure block can be seen in Figure 19. Although CAC1 had the lowest expansion rates in the CAC system group, this cementitious showed the most physical effects. The physical effects can be seen in Figure 20. The expansion carried by ASR is recognized for bringing physical effects such as cracking, it is difficult to attribute the physical effects to the rates of expansion. The damages shown on the upper side of the concrete blocks can be attributed to many factors other than ASR.



Figure 19. CAC1 Concrete Block 5 Sides



Figure 20. CAC1 Physical Damages on Side 5

Figure 21 is the graphical representation of the CSA systems that did not exceed 0.04% to fail ASTM C1293. Only two binders in the CSA system failed the test before 6 months of age by going over 0.06% of the expansion. The cementitious material that experienced the highest expansion rates was PCSA2 but failed the test on day 152. The specimen that reacted the fastest to alkali-silica was CSA-B2 which experienced high expansion rates at day 7. On the other hand, the lowest and more stable expansion generated was fixed to CSA-B1.

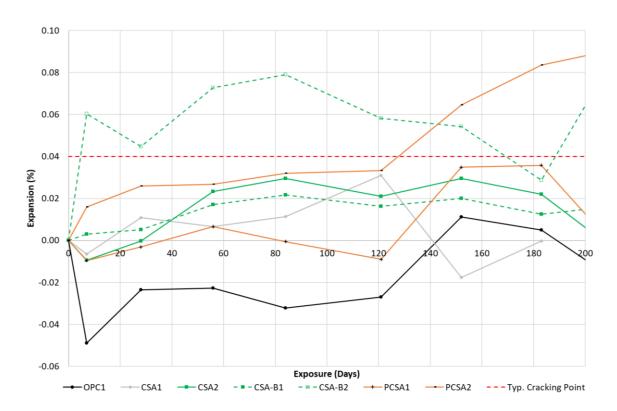


Figure 21. CSA System Expansion Rates

It is still uncertain whether the alkali and chemical composition of each CSA cementitious mixture is directly correlated to ASR. It is a contradictory result that CSA-B1 has the lowest expansion nonetheless, it contains a higher alkali content in relation to CSA-B2. CSA-B1 and CSA-B2 chemical composition is displayed in Table 18.

Table 18. Concrete Exposure Blocks: CSA-B1 and CSA-B2 Chemical Composition

Expansion	ID	Cement Name	<b>Al2O3</b>	CaO	SO3	Na2Oe
Lowest Expansion	CSA-B1	CSA CTS 2	15.82	51.28	16.62	62.22
Highest Expansion	CSA-B2	CSA CTS 3	14.37	53.85	14.40	63.33

The low percentages of expansion of CSA-B1 did not produce any physical signs of ASR as shown in Figure 22.



Figure 22. CSA-B1 Concrete Exposure Block

The high percentages of expansion and early reaction in CSA-B2 generated no physical changes as exposed in Figure 23.



Figure 23. CSA-B2 Concrete Exposure Block

Figure 24 is the graphical representation of the laboratory blended systems that did not exceed 0.04% to fail ASTM C1293. Neither CAC-OPC2 nor CSA-OPC2 surpassed an expansion percentage of 0.03%. Both blended systems experienced similar expansion rate at day 80. The ASR physical effects were not present in either mixture, as shown in Figures 25 for CSA-OPC2 and Figure 26 for CAC-OPC2 respectively.

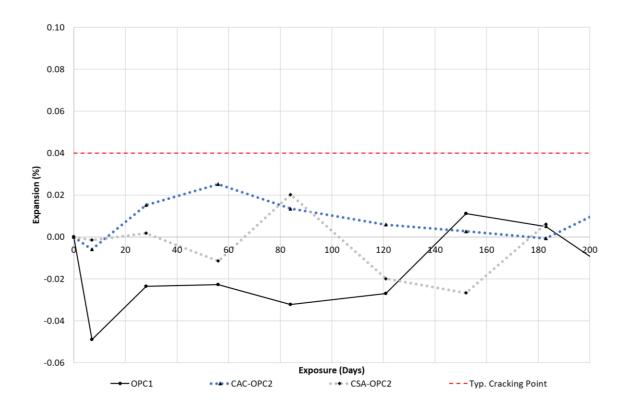


Figure 24. Blended System Expansion Rates



Figure 25. CSA+OPC2 Concrete Exposure Blocks

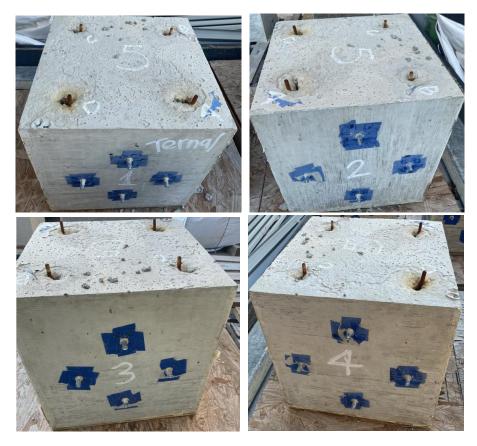


Figure 26. CAC-OPC2 – Ternal NT + Type I/II Concrete Exposure Blocks

# 4.1.1.1 Hydration temperatures and relative humidity outcomes

The peak temperature reached during hydration within the first 7 days of curing, and the lowest temperature reached throughout the 6 months of age for each cementitious material are provided in Table 19. The data available for minimum and maximum temperature for each material was generated by the Hilti sensors. Three out of twelve mixes did not have a sensor installed.

Table 19. Concrete Exposure Blocks: Min and Max Temperatures in Fahrenheit

Minton	Temperature in Fahrenheit					
Mixture	Min	Max				
OPC1	59	128				
CAC1	40	145				
CSA1	45	131				
CSA2	-	-				
CAC-B1	40	100				
CAC-B2	59	134				
CSA2-B1	59	131				
CSA2-B2	59	121				
PCSA1	60	112				
PCSA2	-	-				
CAC-OPC2-B1	-	-				
CSA1-OPC2-B1	46	103				

<sup>-</sup> Data not available, sensor not provided

ASR deterioration results when moisture within the pore solution is absorbed into alkali-silica gel, finalizing in expansive pressure and cracking (Deschenes, 2018). The temperatures in which the concrete specimen is subjected directly affect the moisture content which is correlated to the development of deleterious damages. Out of the 9 cement mixes with data available, CAC1 provided a high temperature surpassing 145°F and CAC-B1 produced the lowest temperature of curing at 100°F at an age as early as 7 days.

Cementitious mixes need to remain moist for them to achieve proper curing.

Typically, the relative humidity needs to surpass 80% for legitimate hydration to occur

(Thomas, 2006). The specimen that showed the least percentage of expansion is CAC1.

The low expansion result can be attributed to a couple of factors. The first factor is given due to the rapid high temperature achieved during the first 7 days of curing.

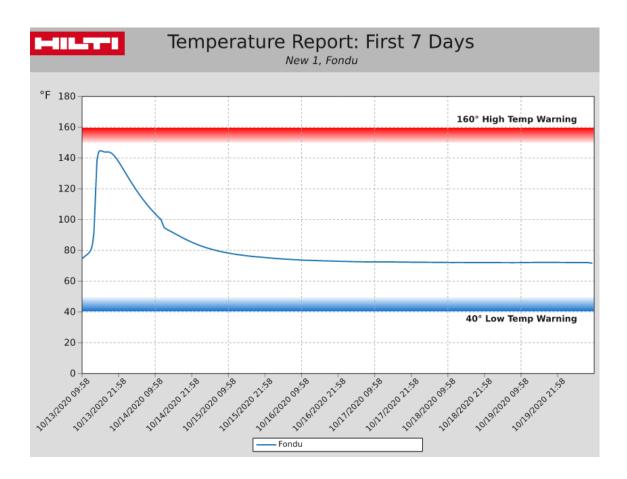


Figure 27. CAC1 First 7 Days Temperature Report Hilti Sensor

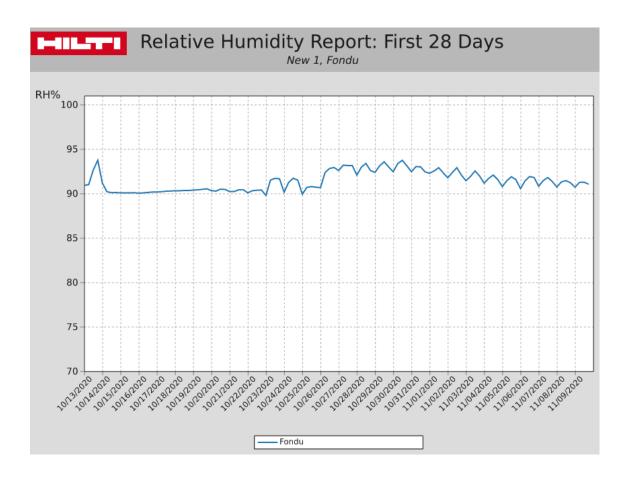


Figure 28. CAC1 First 28 Days Relative Humidity Report Hilti Sensor

In contrast, the CAC-B1 – PP5001 specimen reached a peak temperature of about 100°F during the first 12 hours of hydration. The 100°F peak temperature made CAC-B1 the lowest achiever in the binder. Achieving a lower temperature during curing allows the specimens to be expected to have fewer damages due to ASR. According to the manufacturer of CAC-B1, this type of proprietary cement already contains a certain amount of OPC. Although the amount portland cement contained within the blend is not specified by the manufacturer; the low temperature achieved during curing could be granted to the percentage of portland cement that is not known to be of rapid setting.

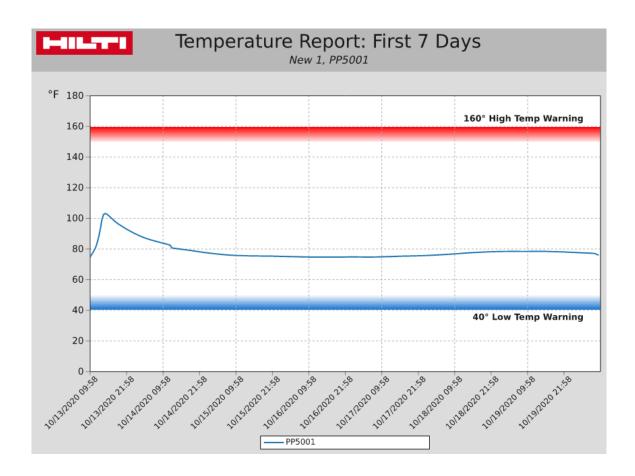


Figure 29. CAC-B1 - PP5001 First 7 Days Temperature Report Hilti Sensor

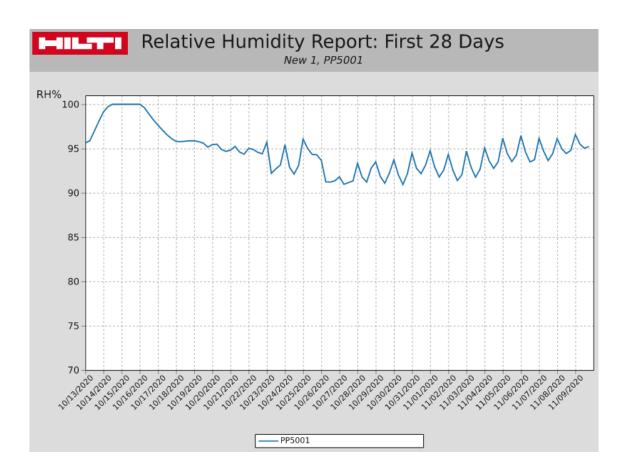


Figure 30. CAC-B1 - PP5001 First 28 Days Relative Humidity Report Hilti Sensor

#### **4.1.2** Concrete Prisms Test (ASTM C1293)

The ASR effects of 3 series of concrete prisms are analyzed in this section. Regarded as the most reliable ASR method, ASTM C1293 manages a 100% relative humidity at 38°C to accelerate the alkali-silica reaction and to identify reactive aggregates within a year of the specimens' age.

Series 1, one was developed using a low W/CM ratio of 0.35. No additional alkalis were used in this series. The expansion averages for the concrete prisms on days 28 through 183 are listed in Table 20. However, at time of writing, OPC1 and CSA2 only achieved 152 days of age.

Table 20. CPT: Series 1 Expansion Averages Days 28 through 183

Mixture	Day							
	28	56	84	121	152	183		
OPC1	0.0032	0.0037	0.0048	0.0051	0.0060	-		
CAC1	0.0064	0.0020	0.0031	0.0033	-0.0044	0.0127		
CSA1	-0.0068	-0.0069	-0.0010	-0.0058	-0.0078	0.0262		
CSA2	0.0014	-0.0009	-0.0018	0.0168	0.0168	-		
CAC-B1	0.0200	0.0780	0.1229	0.1740	0.1809	0.4771		
CAC-B2	-0.0071	0.0390	0.0482	0.0460	0.0465	0.0436		
CSA2-B1	0.0104	0.0206	0.0278	0.0253	0.0250	0.0277		
CSA2-B2	-0.0312	0.0106	0.0182	0.0193	0.0240	0.0298		
PCSA1	0.0022	0.0062	0.0076	0.0100	0.0057	0.0068		
PCSA2	-0.0120	0.0036	0.0038	0.0028	-0.0009	-0.0182		
CAC-OPC2	0.0174	0.0380	0.0438	0.0484	0.0438	0.0464		
CSA-OPC2	0.0166	0.0148	0.0162	0.0177	0.0106	0.0473		

<sup>\*</sup> Measurements given in percent

<sup>-</sup> Indicate data wasn't completed due to recent mixing

Figure 31 allows the perception Series 1 containing the complete 12 mixes' percentage of expansion in respect with age in days.

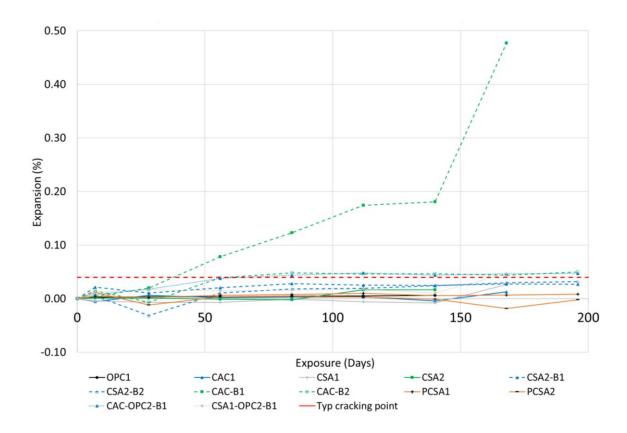


Figure 31. CPT Series 1 – Overall Expansion Rates for CSA, CAC, and OPC

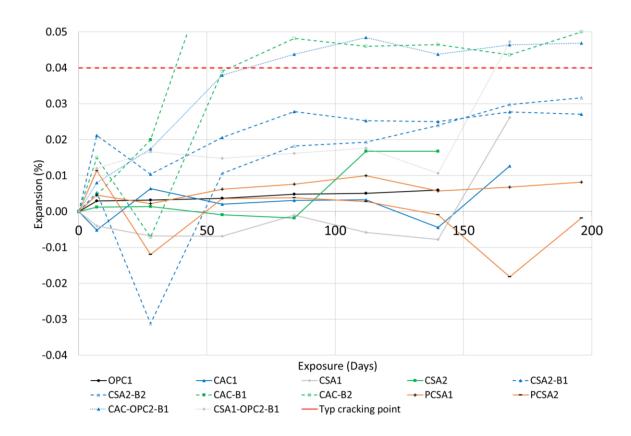


Figure 32. CPT Series 1 – Expansion Less than 0.04%

Thomas (2006) stated that to get ASR to react, it is required to use an appropriate performance test. Currently, there are no tests available to be completely suitable and reliable. Presently, most tests available need the augmentation of the alkali level which led to the development of Series 2 and 3 in this project.

As denoted in ASTM C1293, the value of 1.25% Na<sub>2</sub>O was chosen to accelerate the process of expansion rather reproduce field conditions. Series 1 exposed the most similar mixture design to what is used in the field but kept in the laboratory in a controlled environment.

Series 2 was developed as a modification of ASTM C1293. Series 2 used a low W/CM ratio of 0.35 and additional alkalis of 1.25% with the addition of a 50/50 NaOH. The average percent of the expansion in days 28, 56, 84, 121, 152, and 183 can be acknowledged in Table 21.

Table 21 CPT – Series 2 Expansion Averages Days 28 through 183.

Mixture	Day								
	28	56	84	121	152	183			
OPC1	0.0397	0.1624	0.2413	0.3638	0.3941	0.4417			
CAC1	0.0316	0.0350	0.0429	0.0376	0.0365	0.0346			
CSA1	0.1190	0.2579	0.2950	0.3354	0.3545	0.4032			
CSA2	0.0172	0.0126	0.0162	0.0157	0.0208	0.0195			
CAC-B1	0.1386	0.2058	0.2345	0.2738	0.3067	0.3170			
CAC-B2	0.0371	0.0424	0.0405	0.0459	0.0732	0.2961			
CSA2-B1	0.0450	0.0546	0.0703	0.0849	0.0948	0.0960			
CSA2-B2	0.0352	0.0467	0.0472	0.0472	0.0485	0.0496			
PCSA1	0.0116	0.0688	0.0913	0.1235	0.1425	0.1449			
PCSA2	0.0032	0.0028	-0.0049	-0.0031	-0.0052	-0.0073			
CAC-OPC2	0.0785	0.1846	0.2114	0.2170	0.2182	0.3544			
CSA-OPC2	0.1254	0.2554	0.2974	0.3465	0.3703	0.4245			

<sup>\*</sup> Measurements given in percent

Figure 33 allows the perception Series 1 containing the complete 12 mixes' percentage of expansion in respect with age in days. The control binder OPC1 showed a constant expansion percentage rate throughout the 6 months of age. OPC1 showed the highest expansion, which could be attributed to its 0.71 content of alkali. Although CSAs and CACs were developed to improve OPCs stability, the additional alkalis could have weakened their properties leading them to an early and abrupt expansion rate. There are similarities between the laboratory blended binders and the control binder. OPC1 and laboratory blends CAC-OPC2 and CSA-OPC2 all surpassed 0.3% in expansion.

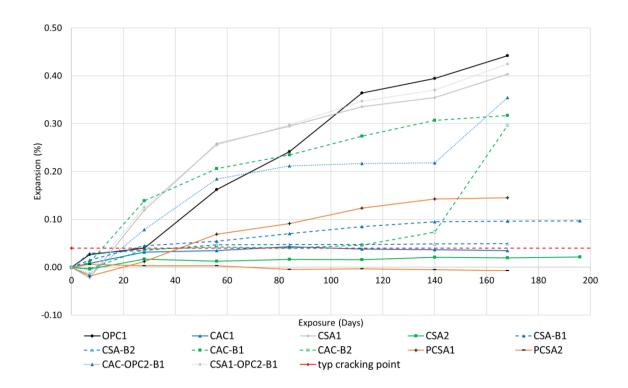


Figure 33. CPT Series 2 – Overall Expansion Rates for CSA, CAC and OPC

Figure 34 shows the specimens that so far pass the CPT test exposing an expansion of less than 0.04%. In Figure 34, it is perceived that PCSA2 experienced some shrinkage after the first 7 days of curing. Initially, it was stipulated that PCSA2 had a dropping percentage of expansions that was related to drying shrinkage as observed in specimens CSA1, CSA2, PCSA1, CAC-OPC2 and CSA-OPC2. It can also be stipulated that the decrease in expansion of PCSA2 could be attributed to an increased rate of alkali leaching. However, it is still unclear the reason for the rate of expansion to keep dropping after the curing and drying phase.

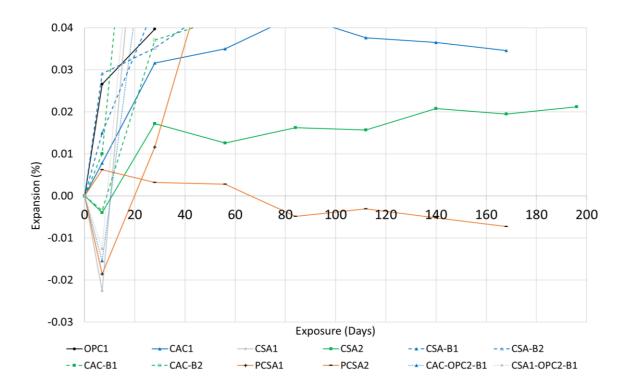


Figure 34. Series 2 Binders Tending to Pass CPT Under 0.04%

Series 3 followed the standard ASTM C1293 specification. Series 3 contained a total additional alkali content of 1.25% with the addition of a 50/50 NaOH solution. Diamond (1997) expressed the paradox of how adding alkali hydroxide to cements to raise the alkali level in ASR experiments can be an uncertain practice that can lead to the cause of unforeseen consequences. In Series 3, most of the binders appear to maintain a percentage of expansion lower than 0.5% until day 140.

Table 22. CPT – Series 3 Expansion Averages Days 28 through 183

Mixture	Day								
	28	56	84	121	152	183			
OPC1	0.0596	0.1762	0.2156	0.2286	0.2628	0.3740			
CAC1	0.0133	0.0219	0.0177	0.0196	0.0173	0.0295			
CSA1	0.0133	0.0187	0.0155	0.0348	0.2211	3.8155			
CSA2	0.0333	0.0348	0.0291	0.0301	0.0281	1.1136			
CAC-B1	0.0820	0.1378	0.1777	0.2748	0.2276	0.6685			
CSA2-B1	0.0505	0.0727	0.0780	0.0823	0.0853	0.8165			
CSA2-B2	0.0083	0.0116	0.0053	0.0039	0.0043	0.0433			
PCSA1	0.0156	0.0319	0.0509	0.0629	0.0667	0.2149			
PCSA2	0.0123	0.0133	0.0076	0.0079	0.0060	0.0327			
CAC-OPC2	0.0976	0.1448	0.1731	0.1964	0.2276	1.3624			
CSA-OPC2	0.1272	0.2987	0.3539	0.3744	0.4051	0.3930			

<sup>\*</sup> Measurements given in percent

The only binders that maintained an expansion of less than 0.04% over 180 days were PCSA1, CSA-B1, CSA-B2, Being the control binder with a 0.72 content of Na2Oe, OPC1, failed the test in the first 30 days. Typically, the higher W/CM ratio causes the lower strength. The lower strength can cause the ASR to be accelerated.

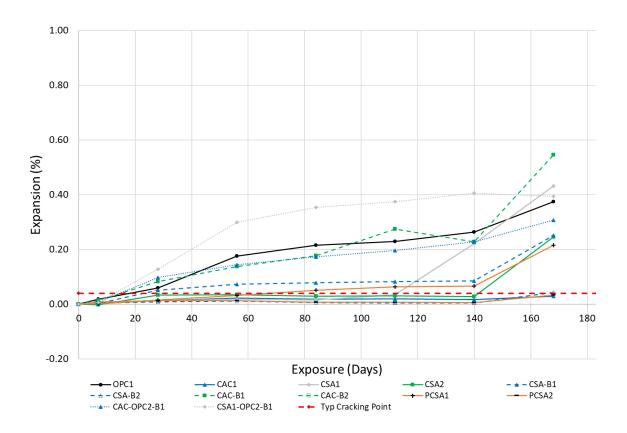


Figure 35. CPT Series 3 – Overall Expansion Rates for CSA, CAC and OPC

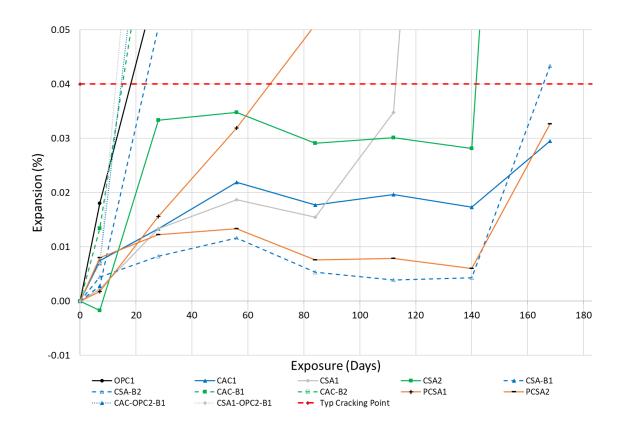


Figure 36. CPT Series 3 – Expansion Less than 0.04%

To get a stronger understanding of the ASR effects on straight and blended cements, the straight mix CSA1 and blended mix CSA1-OPC2 are analyzed and shown in Figures 37. Figure 37 also shows the minimal effects on the prisms prepared with a blend of 25% CSA1 and 75% OPC2. Figure 38 shows the ultimate major cracks in the prisms from the straight mix of cement CSA1. It can be deduced from the observations that the blended system gave the stability of the specimen to maintain the structural components almost intact until day 183 of age.



Figure 37. CSA1 25% - OPC2 75% (2) Cracks on CSA1 25% Specimen B

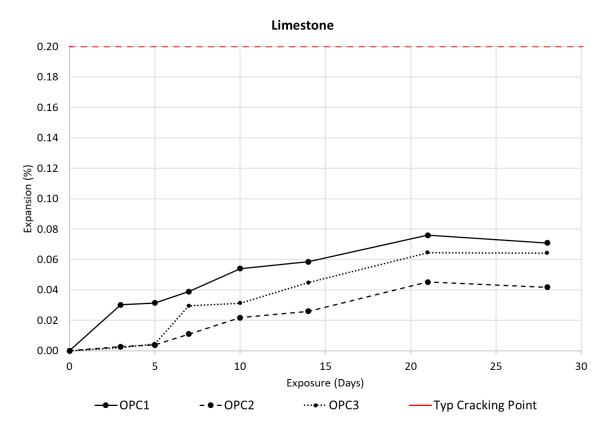


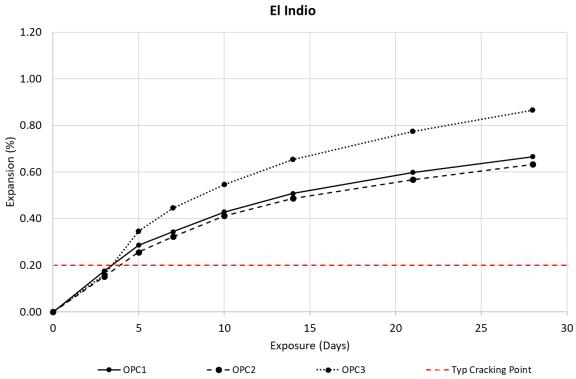
Figure 38. (1) CSA1 100% Specimen B on Measurement Apparatus (2) CSA1 Specimen B Cracks (3) CSA1 Specimen C on Apparatus (4) CSA1 Specimen C Cracks

### **4.1.3** Accelerated Mortar Bar Test (ASTM C1260/C1567)

According to ASTM C1260, the specimens should fail the test if passing 0.20% in expansion rates. The results obtained from the Accelerated Mortar Bar were analyzed per type of cement. The cements were divided into OPC, CSA, CAC, CSA blends, and CAC blends. The cements were analyzed in relation to the type of aggregate. The aggregates used for this series were highly reactive Jobe, moderately reactive El Indio, and nonreactive Limestone.

Figure 39 show the expansion of the OPC specimens. It is observed that Jobe OPC specimens had the most expansion. All the OPC specimens under Jobe failed the test on day 3. OPC3 with Jobe had the highest expansion of 0.96%, whereas OPC3 with nonreactive Limestone only reached an expansion of 0.06%. None of the OPC specimens using Limestone failed the test. The OPC specimens with El Indio had a moderately accelerated expansion and failed ASTM C1260 on day 5.





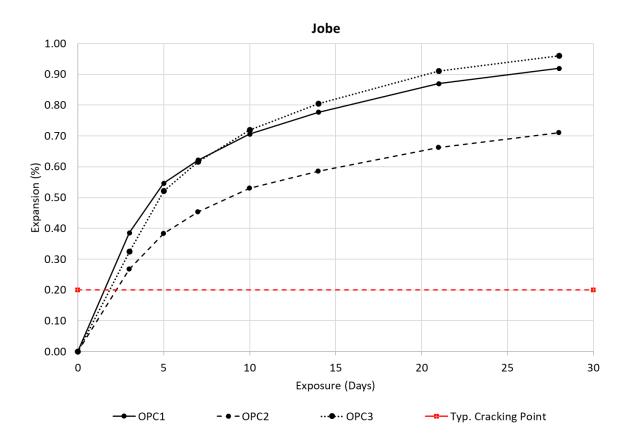
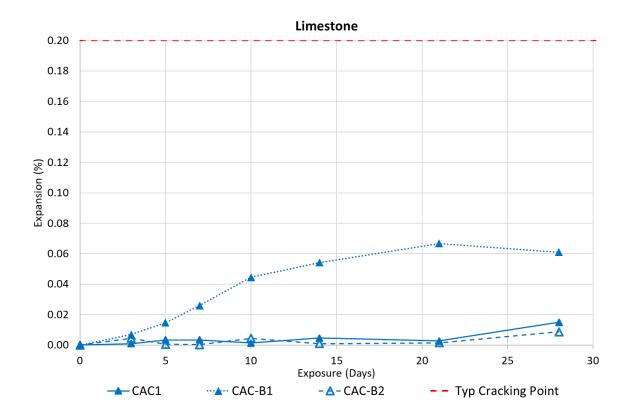
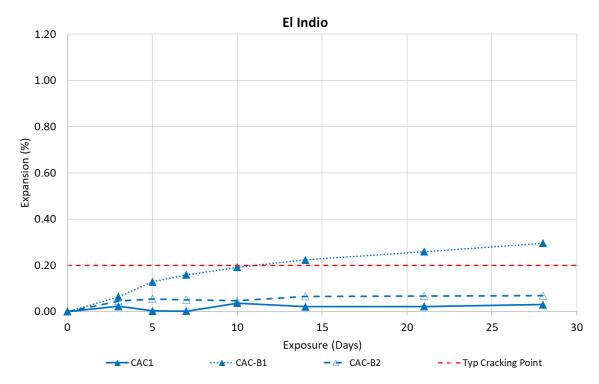


Figure 39. OPC – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion

The expansion rates of the CAC specimens in respect to Jobe, El Indio and, Limestone can be seen in Figure 40. It is observed that Jobe CACs specimens had the most expansion. Only CAC-B1 with Jobe failed the test on day 3. The moderately reactive aggregate El Indio delayed the expansion of CAC-B1. Specimen CAC-B1 with El Indio failed the test on day 10. None of the limestone CAC specimens failed the test nor surpassed an expansion of 0.07%. The results of CAC-B1 expansion on Jobe, El Indio and Limestone indicate that CAC-B1 is a reactive aggregate regardless of the type of aggregate being used within the mix. On the other hand, it can be deduced that CAC1 is not deleterious even when using highly reactive aggregates.





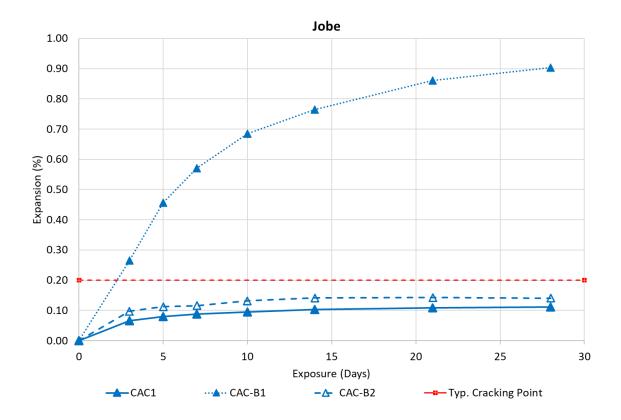
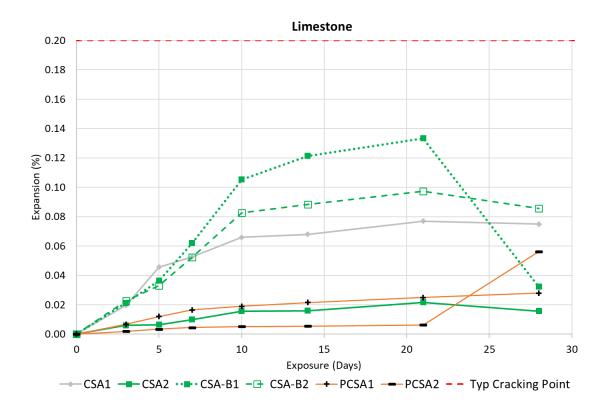
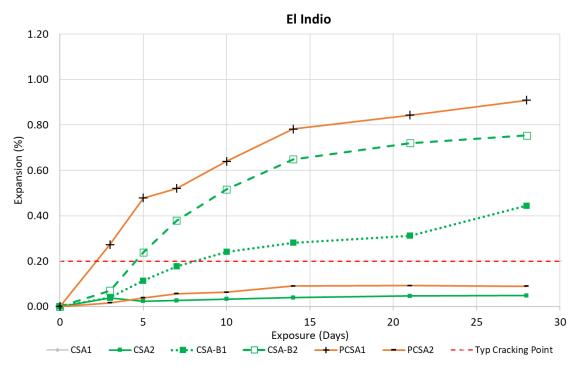


Figure 40. CAC – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion

The expansion rates of the CSA specimens in respect to Jobe, El Indio, and Limestone can be observed in Figure 41. Neither of the CSA specimens had an expansion surpassing 0.20% when being mixed with Limestone. However, a specimen that had a relatively high expansion rate in respect to all three aggregates was CSA-B1. CSA-B1 failed the test at day 10 when using moderately reactive El Indio but failed the test on day 7 when using the considered very reactive Jobe.





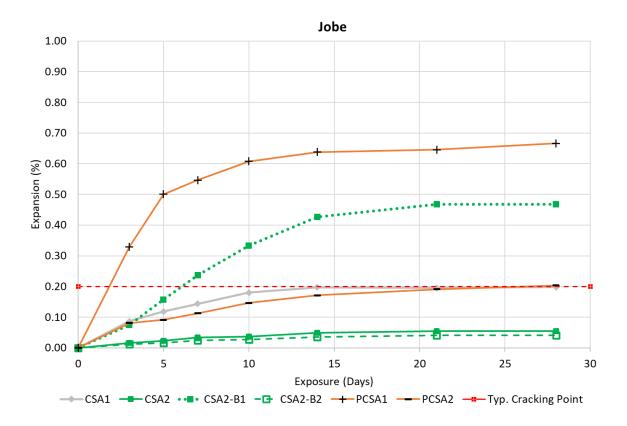
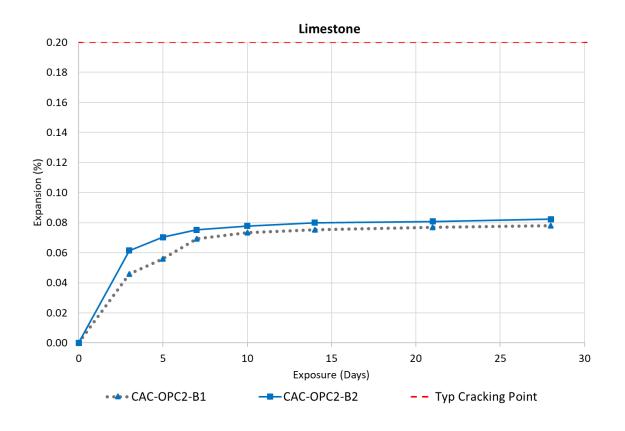
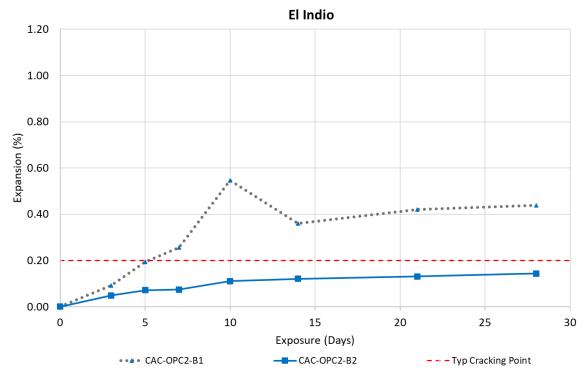


Figure 41. CSA – Limestone, El Indio, Jobe Mortar Bar Test Overall Expansion





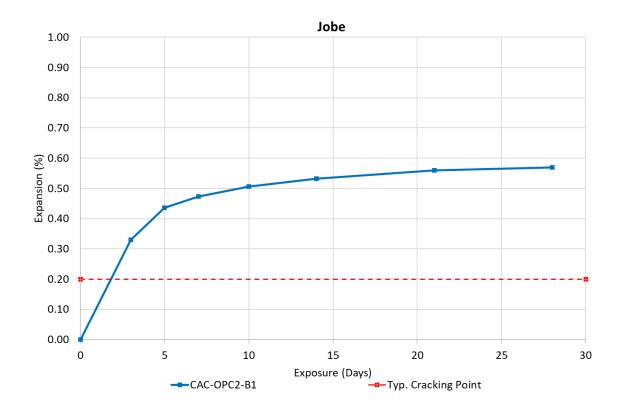
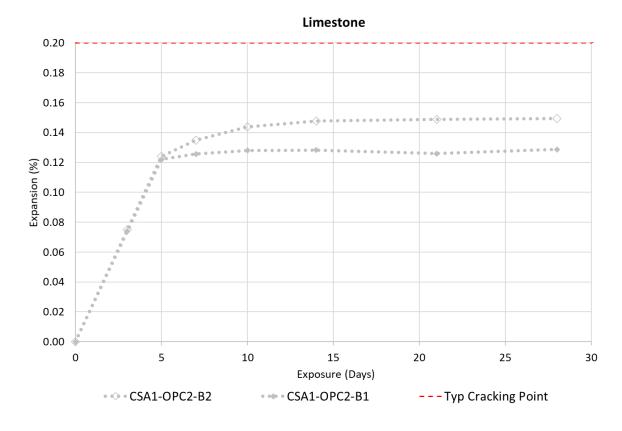
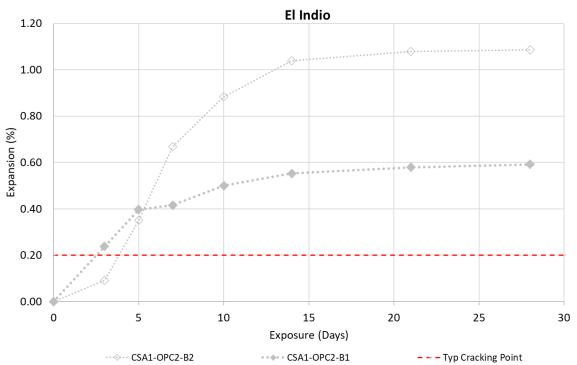


Figure 42. CAC/OPC2 blends – Limestone, El Indio, Jobe Overall Expansion





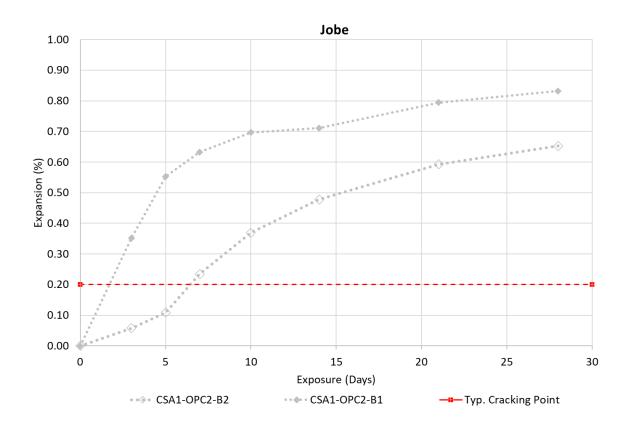


Figure 43. CSA/OPC2 blends – Limestone, El Indio, Jobe Overall Expansion



Figure 44. (1) El Indio, CSA1 75% - OPC2 25% (2) Back View of Prism (3) Cracks on Prisms

It was observed from CAC, CSA, OPC, and blended binders that the expansion rates did increase when mixing with the very highly reactive aggregate Jobe. Therefore, the outcomes from this test confirmed the correlation between the reactivity level of the aggregate with the type of cement. When comparing OPC, CAC, CSA, and blender binders; CSA-OPC blends showed the most susceptibility to ASR expansion when using Limestone, El Indio, and Jobe. None of CSA-OPC failed the test when using Limestone. CSA1-OPC2-B2 which used only 25% of CSA1 and 75% of OPC2 kept a higher expansion in respect to CSA1-OPC2-B1 which used 75% of CSA1 and 25% of OPC2.

## **4.2** Delayed Ettringite Formation

### **4.2.1** Accelerated High Temperature Curing (Kelham Test Method)

Currently, there are no standard ASTM or AASHTO test methods to obtain the possible effects of DEF in cementitious materials in a laboratory setting. The contributions of DEF are difficult to separate from the effects of distinct mechanisms such as ASR. However, two methods had been using to drive to conclusive reasoning in this project. The methods using were the Kelham Test Method and the Modified Test Method. This project applied the reasoning from Kelham (1996) to cover molds with a water-soaked cloth to ensure 100% RH during curing. Samples were maintained at 23°C then subjected to a heat curing cycle. The curing regime according to the Kelham method reached 95°F and the Modified Method peaked at 65°F. Managing the same cements and maintaining the same aggregate Limestone, The Kelham and The Modified method were addressed to accelerate the means in which DEF could affect the cementitious material.

The Kelham Method specified that DEF can only occur when drying the specimens at 65°C in a moist environment. Table 24 expresses the Kelham Method Series Expansion Rates. Binders exposed to the Kelham method curing regime obtained far more expansion percentages than the ones exposed to the Modified Method.

Table 23. DEF – Kelham Method  $95^{\circ}F$  Expansion Rates Day 0 through 183

N#: 4	Day							
Mixture	0	7	28	56	84	121	152	183
OPC1	0.0000	0.0230	0.0383	0.0148	0.0475	0.1730	0.3670	1.1100
OPC2	0.0000	0.0200	0.0348	0.0475	0.0893	0.0055	0.2998	0.3713
OPC3	0.0000	0.0215	0.0363	0.0545	0.2120	1.1883	1.2505	1.3083
CAC1	0.0000	0.0135	0.0168	0.0103	0.0153	0.0185	0.0160	0.0193
CSA1	0.0000	0.0208	0.0215	0.0230	0.0310	0.0593	0.0588	0.0558
CSA2	0.0000	0.0185	0.0903	0.0168	0.0225	0.0255	0.0240	0.0265
CAC-B1	0.0000	0.0610	0.1420	0.0758	0.1230	0.1813	0.4558	0.4935
CAC-B2	0.0000	0.0445	0.1203	-	-	-	-	-
CSA2-B1	0.0000	0.0170	0.0205	0.0130	0.0203	0.0248	0.0213	0.0248
CSA2-B2	0.0000	0.1330	0.1450	0.1423	0.1527	0.1697	0.1690	0.1637
PCSA1	0.0000	0.0393	0.0201	0.1021	0.0960	1.1710	1.2345	1.3375
PCSA2	0.0000	0.0290	0.0307	0.0323	0.0323	0.0340	0.0323	0.0407
CAC-OPC2-B1	0.0000	0.0205	0.0218	0.0215	0.0220	0.0230	0.0540	0.1320
CSA1-OPC2-B1	0.0000	0.0343	0.0563	0.2535	0.6900	1.2718	1.9035	2.0380

<sup>\*</sup> Measurements given in percent

<sup>-</sup> Indicate data wasn't completed due to recent mixing

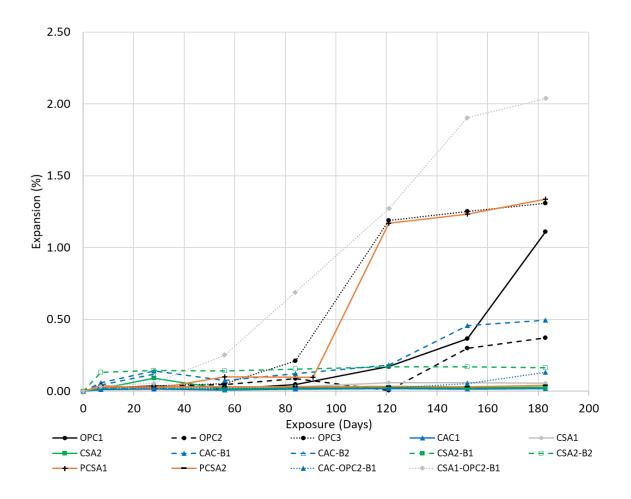


Figure 45. Kelham Method 95°F Peak Series Expansion with Time

By far, the reasoning exposed by Kelham and Fu that DEF expansion only occurs when putting specimens through temperatures higher than 65°F can be observed in this project. The Modified method managed a peak temperature of 65°F and none of the cementitious materials surpassed 0.30% of expansion. The contrast of this analysis is to compare to the Kelham method where 4 out of 14 mixes surpassed 1.0% of expansion on day 183.

In both series, the formation of ettringite was clearly higher in the laboratory blended systems. The blended binder CAC-OPC2-B1 which used 75% of CSA1 and 25% of CSA1 maintained the highest expansion rate of expansion in both Kelham and

Modified Method. For laboratory blended cements, hardening seemed to occur slower since OPC reacts only during the first days regardless of the temperature of curing.

Ye'limite hydration occurred in the presence of lime and formed ettringite faster than in the rapid hardening systems.

Table 24. DEF – Modified Method 65°F Expansion Rates Day 0 through 183

Mixture	Day							
	0	7	28	56	84	121	152	183
OPC1	0.0000	0.0130	0.0213	0.0240	0.0333	0.0170	0.0188	-
OPC2	0.0000	0.0058	0.0080	0.0070	0.0073	0.0065	0.0073	-
OPC3	0.0000	0.0098	0.0158	0.0125	0.0130	0.0100	0.0128	-
CAC1	0.0000	0.0408	0.0758	0.0905	0.0958	0.1003	0.1293	-
CSA1	0.0000	0.0128	0.0245	0.0295	0.0298	0.0283	0.0223	1
CSA2	0.0000	0.0033	0.0010	0.0050	0.0078	0.0075	0.0080	-
CAC-B1	0.0000	0.0075	0.0105	0.0135	0.0150	0.0175	0.0193	-
CAC-B2	0.0000	0.0373	0.0653	0.1440	0.1623	0.1677	0.1733	-
CSA2-B1	0.0000	0.0098	0.0138	0.0253	0.0278	0.0270	0.0280	1
CSA2-B2	0.0000	0.0073	0.0103	0.0120	0.0133	0.0110	0.0043	-
PCSA1	0.0000	0.0188	0.0193	0.0240	0.0283	0.0388	0.0515	-
PCSA2	0.0000	0.0010	0.0020	0.0017	0.0187	0.0197	0.0163	-
CAC-OPC2-B1	0.0000	0.0013	0.0020	0.0078	0.0425	0.0418	0.2520	-
CSA1-OPC2-B1	0.0000	0.0290	0.0320	0.0298	0.0365	0.0355	0.0273	-

<sup>\*</sup> Measurements given in percent

<sup>-</sup> Indicate data wasn't completed due to recent mixing

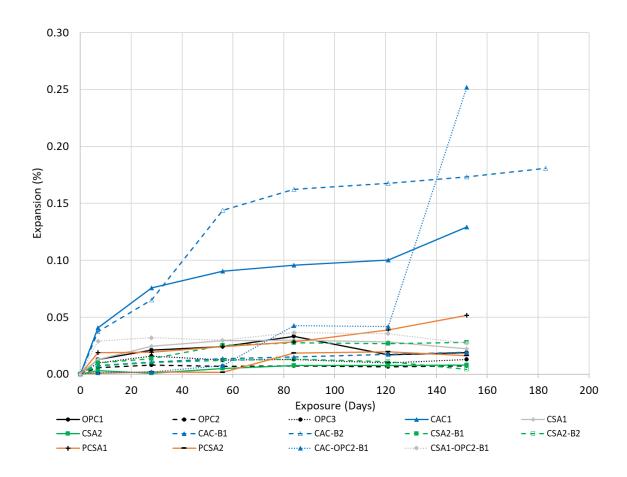


Figure 46. Modified Method 65°F Peak Series Expansion with Time

#### 5. CONCLUSIONS AND RECOMMENDATIONS

# 5.1 Durability performance

This thesis attempted to achieve a better understanding of the reactivity of alkalis and sulfates on RSHCs with respect to ASR and DEF. It provided a better understanding of the mechanisms that CAC, CSA, and laboratory blended binders have with respect to ASR and DEF. The conclusive reasoning of the influence of the standardized and modified performance test methods on RSHCs related to ASR and DEF were analyzed in short-term and long-term data.

## Concrete Exposure Blocks:

- Regarding the concrete exposure block testing and conclusion, it is recommended
  to complete the two-year expansion to make definite and reliable statements. The
  alkali content of each cement cannot be attributed to be directly correlated to ASR
  expansion rates.
- 2. Comparing OPC, CSA, CAC, and blended binders leads to the conclusion that blended binders CAC-OPC2 and CSA-OPC2 managed the least expansion rates throughout the test. In contrast, the highest expansions were given to CSA proprietary cements. It is known that CSA proprietary cements contain certain amount of OPC. Therefore, the conclusion of OPC being a possible factor affecting the integrity and incrementing ASR reactivity of the specimens can be stated.
- 3. Chemical responses go beyond complicated in outdoor concrete specimens, therefore, there were no assumptions or reliable conclusions yet made. Making

assumptions of the reactivity observed in this study might be incorrect and misleading. However, the observance of different influencing factors such as, weather, temperature, sun exposure, were granted to the level of expansion or shrinkage of each block.

#### Concrete Prisms:

- CACs managed a relatively higher ASR reactivity in comparison to OPC, CSA, and blended binders. The only specimen that surpassed 0.04% expansion at day 56 was CAC-B1, whereas, specimens such as PCSA2 did not surpass 0% of expansion and experienced a high level of drying shrinkage.
- 2. The alkali augmented managing a low w/cm ratio, Series 2, concluded with 9 out of 12 mixes failing the test at as early as 28 days of age. The addition of alkalis increased the reactivity in Series 2 reaching expansions higher than 0.40%.
- 3. The specimens that did not react to the augmentation of alkalis in Series 2 were CAC1, CSA2 and, PCSA2. This observation can deduce that CSAs are less susceptible to ASR reactivity even when containing additional content of alkalis.
- 4. Series 3 managed a mix design exactly according to ASTM C1293. Series 3 mix design contained an additional amount of alkalis and a higher w/cm ratio. Per data information, Series 3 specimens had a more linear progressive expansion rate which increased at day 140.
- 5. Only 3 specimens out of 12 specimens in Series 3 seemed to have passed the CPT test. At the time of writing, these specimens had only 160 days of age. Although these specimens, PCSA2, CSA-B2, and CAC1, did not fail the test up to day 160;

they are expected to fail the test by day 183. This observation can deduce that CSAs are less susceptible to ASR reactivity even when containing additional content of alkalis and a high w/cm ratio.

#### Accelerated Mortar Bar:

- It was determined that the non-reactive aggregate Limestone led neither OPC1,
   OPC2 nor OPC3 to surpassing 0.08% in expansion.
- 2. The use of Limestone, El Indio, and Jobe did influence the expansion of OPC2. The results confirm that OPC2 is the cement that manages the least reactivity even when using highly reactive aggregates. OPC2 maintained the lowest expansion throughout all aggregates in comparison to OPC1 and OPC3.
- 3. All specimens failed the test at day 3 when mixed with very reactive Jobe sand. The use of the moderately reactive specimen El Indio allowed OPC1, OPC2, and OPC3 to delay the release of alkalis that generate expansion and to fail the test at day 5.
- 4. The CAC specimen that showed the most sensitivity to expansion due to ASR was CAC-B1. Yet, the expansion of CAC-B1 did not lead to a strong enough reactivity to generate expansion surpassing 0.08% when using non-reactive Limestone.
- 5. CAC-B1 was highly influenced by the reactivity of the aggregate which leads to surpassing 0.90% in expansion when using very reactive Jobe but only 0.40% when using moderately reactive El Indio. Neither CAC-B2 nor CAC1 failed the test even when mixed with highly and moderately reactive aggregates.

6. When comparing OPC, CAC, CSA, and blender binders; CSA-OPC blends showed the most susceptibility to ASR expansion when using Limestone, El Indio, and Jobe. None of CSA-OPC failed the test when using Limestone. CSA1-OPC2-B2 which used only 25% of CSA1 and 75% of OPC2 kept a higher expansion in respect to CSA1-OPC2-B1 which used 75% of CSA1 and 25% of OPC2.

### **DEF**:

- 1. The reactivity expansion values observed in the Modified Method confirm the hypothesis that DEF expansion only occurs when putting specimens through temperatures higher than 65°F when the cement paste hydration.
- There was not a significant rate of expansion in the Modified Method due to the low temperature exposure that didn't activate the development of delayed ettringite.
- The blended binder CAC-OPC2-B1 which used 75% of CSA1 and 25% of OPC2
  maintained the highest expansion rate of expansion in both Kelham and Modified
  Method.
- 4. For laboratory blended cements, hardening seemed to occur slower since OPC reacts only during the first days regardless of the temperature of curing. Ye'limite hydration occurred in the presence of lime and formed ettringite faster than in the rapid hardening systems.

#### 5.2 Future work

The concrete exposure block, ASR concrete prism and DEF mortar bar specimens will remain to be carefully monitored and measured to reach the end of the studies at 48

months of age. Long-term data to define quality and durability of RSHCs in respect to ASR and DEF will continue to be analyzed to obtain proof that the testing and materials are suitable for construction. With the use of further studies and modified versions of the standard tests the performance of the CSA and CAC cements will better prepared for use.

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