

AN ACCELERATED TEST METHOD TO EVALUATE CEMENTITIOUS  
MIXTURES SUBJECTED TO CHEMICAL SULFATE ATTACK

by

Sushmitha Mercy Joseph Prabhakar, B.Tech

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Committee Members:

Federico Aguayo, Chair

Yoo-Jae Kim

Anthony Torres

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## **DEDICATION**

I would dedicate my thesis work to Jesus Christ my Savior and the solid rock on whom I stand, for his unfailing grace.

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## 1. INTRODUCTION

Altogether for a concrete structure to be intended for its most extreme service life, cautious thought must be made when assessing the performance of a concrete blend for its expected condition. This is particularly valid for concrete exposed to sulfate-bearing soils, ground water or seawater where external sources of sulfate can enter the cement matrix and lead to extreme disintegration of the structure. Commonly known as chemical sulfate attack, this type of concrete deterioration has been known to diminish the long-term strength and durability of concrete structures. Damage of concrete because of chemical sulfate attack is an extensive process that is impacted by numerous elements, including but not limited to the type of cement, type and concentration of sulfate solution, and due to the groundwater conditions (i.e., stagnant versus running water). Depending upon the severity of the exposure conditions decided from sulfates present in the soil or water, ACI 201.2R - Guide to Durable Concrete, gives suggestion to relieve harm from external sulfate attack dependent on constraining cementitious blend parameters, for example, water-to-cementitious proportions (W/CM), compressive strength, and cementing materials (Guide to Durable concrete, ACI, 1992). Prescriptive prerequisites, in any case, regularly neglect to take after the solidness attributes of current cement and upset the utilization of modern and advanced concretes. ACI allows the use of other combinations of cementing materials in sulfate exposure, provided that performance testing utilizing ASTM C1012-Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution (mb, Thomas, Folliard & Drimalas, 2013) demonstrates that the expansion limit for the appropriate exposure class is not exceeded. The technique quickens the attack mechanism by inundating mortar bars in an aggressive

sodium sulfate solution (5% Na<sub>2</sub>SO<sub>4</sub>) and observing the length change. The test frequently requires measurements that may last from a half year to a year. Thus, making it an exceptionally disagreeable for concrete producers to wait that long. Therefore, new quickened lab tests are required that more precisely reflect long-term durability and performance for concrete under sulfate exposure, while acquiring results within a relatively short duration of time. In this study a new proposed test strategy is developed that fundamentally diminishes the length of the ASTM C1012 test by quickening the penetration and dissemination of sulfates (SO<sup>-</sup>) in mortar samples set under high vacuum. This study assesses the presentation of standard mortars cast utilizing a blend of cementitious materials and evaluated using a new performance method which compares and analyzes three different test methods for determining sulfate resistance. Since the choice of the degradation measure may lead to different conclusions, regarding the relative performance of concrete types, one single measure may not suffice to characterize the degradation sufficiently. Therefore, in this study was recommended to use multiple relevant indicators to investigate the resistance of concrete against sulfate attack.

## **1.1 Background**

External sulfate attack continues to be a significant risk to the long-term performance of concrete structures. Soil or water containing adequate degrees of sulfates can enter and chemically respond with the cement hydrates, prompting volumetric expansion, and in serious cases, mellowing of the cement matrix. While ASTM C1012 is the most broadly acknowledged execution test method used to determine the sulfate resistance of cementitious systems, the test requires in any event a half year, and

frequently a year to perform. Consequently, methods to mitigate or prevent harm from external sulfate attack in new concrete structures have been dependent on prescriptive needs.

A few researchers, (Dhole, 2008), (Drimalas, 2007), (mb, Thomas, Folliard & Drimalas, 2013), (ASTM C109/C109M-16a, 2018). have indicated that the utilization of suitable cement and supplementary cementitious materials (SCMs), as well as minimizing permeability using a low w/c, can demonstrate satisfactory performance for concrete exposed to sulfate-rich environments. The use of a sulfate-resistant Portland cements such as Type II or Type V can reduce the severity of attack by limiting the tricalcium aluminate ( $C_3A$ ) available to react and form secondary ettringite, which is the phase that causes expansions when formed in small pores within a certain size range (Tian & Cohen, 2000). As such, so as to control the obstruction against sulfate attack, ASTM C150 constraints the  $C_3A$  content for sulfate resistant cements to 8% and 5% for Type II and Type V, respectively.

In previous study done by (Aguayo, 2019) (Aguayo, Drimalas & Folliard, 2019), the vacuum impregnation technique was used which indicated a vast improvement in time to failure when compared with the ASTM C1012. The proposed test method showed a significant acceleration in the rate of expansion with severe visual deterioration observed two to three times sooner. While visual degradation and measured expansion occurred more rapidly for specimens subjected to the accelerated method and immersed in 5 %, samples still showed much faster expansion when immersed in a less aggressive 0.89 %  $Na_2SO_4$  solution than companion specimens tested according to ASTM C1012 in 5 %  $Na_2SO_4$  (Aguayo, Drimalas & Folliard, 2019). The proposed research presented

herein uses a similar technique however, specimens are also placed in a pH-controlled environment which further accelerates the rate of expansion observed in mortar specimens. In this study, samples were all monitored for their expansion over time from sulfate exposure using length change measurements in addition, specimens were also evaluated for their compressive strength change pre- and post-immersion in sulfate solution to assess performance.

## **1.2 Research Significance**

This new proposed test method is aimed at significantly reducing the duration of the ASTM C1012 by accelerating the diffusion of sulfates in mortar specimens placed under high vacuum and by exposing it to a pH-controlled environment. Moreover, a variation in the chemistry by using different fly ashes and different types of cement makes the test more optimized by providing a wider range of test results to compare.

## **1.3 Hypothesis Statement**

The following hypothesis for the research study were drawn:

- **Hypothesis:** Controlling the pH of the test solution will accelerate the rate of sulfate degradation and subsequent expansion in mortar specimens.
- **Null hypothesis:** Controlling the pH of the test solution will not accelerate the rate of sulfate degradation and subsequent expansion in mortar specimens.

## **1.4 Objective of Research**

The following objectives were set in place to achieve the research scope previously presented:

1. To reduce the duration of the ASTM C1012 by accelerating the penetration and diffusion of sulphate's in mortar specimens placed under high vacuum AND exposing to a pH-controlled environment.
2. The analyse and assess sulfate performance of mortar bars and cubes pre- and post-sulfate exposure through length and compressive strength change, respectively.
3. Evaluate and access performance on a range of mixtures varying in chemistry including different fly ashes and types of cement.

## **1.5 Thesis Organization**

The first chapter of this report is the introduction. It is comprised of a background discussion on sulfate attack on concrete, review of the statement of problem, the objective of this research, followed by the significance of this research and organization of this research report. The second chapter of this report presents a literature review on sulfate attack background, types of sulfate attack, steps to mitigate sulfate attack followed by the field performance and the influence of sulfate attack. The third chapter is various methodologies involved on the study namely the controlled method/standardized method (ASTM C1012), vacuum impregnated method and the pH-controlled method along with the materials and procedures involved. The fourth chapter of this report presents the length change of hydraulic cement motors and the expansion across all the three test

methods. Like the fourth chapter five also talks about the compressive strength of hydraulic cement mortars involving the compressive strength change across all the three tests. The chapter six discusses about the influence each test method has on the mortar bars. Finally, the seventh section gives the overall conclusions for this research based on the lab findings.

## **1.6 Assumption, Limitations, Delimitations**

Several assumptions were necessary during the research work. Firstly, it was assumed that the vacuum chambers should perform the same as the study done by (Aguayo, 2019) which was done using slightly different vacuum chambers; However, the vacuum pressure and procedures were kept identical. Secondly, it was assumed that some level of strength loss that would be considered detrimental for the service life of a structure and thus, an indication that sulfate attack is progressing and leading to degradation in our study. For instance, ASTM C1012 method suggests failure of the mortar bars is defined as a length change exceeding 0.10 % expansion or fracture. Thus, for the strength loss of the specimen we intend to determine what value would suggest failure in the performance of the structure. Also, suggesting an improvement to the current method by determining which one of the three methods is the most accelerated thereby saving time. Finally, it was assumed that all specimens would perform equally regardless of specimen type. In other words, regardless of mortar cube or prism they would perform the same in terms of degradation (cracking, expansion, scaling, etc.) based on similar exposure conditions (i.e., static submerge, vacuum impregnated, and pH controlled).

The work conducted within this research was limited to only a 5% sodium sulfate solution. Although there are a variety of sulfate types and concentration found in the field, the scope of this research was limited to only 5%. In addition, the research was also limited to a relatively small tank size for the pH-controlled system due to lab space. Consequently, this also limited the study to a certain number of mixtures. Lastly, it is also worth mentioning the limitation in the time constraint involved in completing the test. All the 12-14 months data for the samples were not collected for all the mixes, in particular the mix 8 only had data for 8 weeks due to the lab construction and COVID-19 pandemic which resulted in our lab being shut down for a period.

The research also included delimitations such as the storage of the specimens and the temperature at which the specimens were cured prior to sulfate exposure. As oppose to following the same procedure according to the ASTM 1012 standard in which the specimens are heated for 24 hours at 38°C and once 20 MPa strength is reached the specimens are tested, a standard curing of 50°C for 7-days was used across all mixtures and testing methods. Lastly, the length of the experiment i.e., the intervals at which the specimens are measured was controlled and remained uniform for all the mixes.

## 2. LITERATURE REVIEW

### 2.1 Sulfate Attack

#### 2.1.1 Sulfate Attack Background

Sulfate attack is a form of concrete deterioration which is very common. It occurs when concrete meets water containing sulfates ( $\text{SO}_4$ ). Sulfates can be found in some soils (especially when arid conditions exist), in seawater, and in wastewater treatment plants. Altogether for a concrete structure to be intended for its most extreme service life, cautious thought must be made when assessing the performance of a concrete blend for its expected condition (Guide to Durable concrete, ACI, 1992). This is particularly valid for concrete exposed to sulfate-bearing soils, ground water or seawater where external sources of sulfate can penetrate and lead to extreme disintegration of the cement matrix. The chemical sulfate attack has been known to diminish the strength of concrete structures. Deterioration of concrete because of chemical sulfate attack is an extensive process that is impacted by numerous elements, including but not limited to the type of cement, concentration of sulfate solution, and due to the groundwater conditions (i.e., stagnant versus running water). Waterborne sulfates can react with hydration products such as the tri-calcium aluminate ( $\text{C}_3\text{A}$ ) phase of Portland cement, and with calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) to form calcium sulfate (gypsum) and then on further reaction forms ettringite. Expansion due to ettringite formation causes tensile stresses to develop in the concrete (Guide to Durable concrete, ACI, 1992). Once these stresses become greater when compared with the concrete's tensile capacity, the concrete tends to crack. These cracks provide easy ingress for more sulfates into the concrete and accelerating the

deterioration. Depending on the severity of exposure conditions determined from sulfates measured in the soil or water, ACI 201.2R, Guide to Durable Concrete, provides recommendation to mitigate damage from external sulfate attack based on limiting mix design parameters such as water-to-cement ratios (w/c), compressive strength, and cementing materials (Guide to Durable concrete, ACI, 2016).

### **2.1.2 Types of Sulfate Attack**

While there are numerous studies and complexities surrounding around sulfate attack, the scientific community has generally recognized two types of deleterious deterioration by sulfate: internal and external sulfate attack. This section presents a detailed summary of previously published literature on “classical” chemical sulfate attack however, a brief introduction on internal is also included.

#### ***2.1.2.1 Internal sulfate attack***

Internal sulfate attack occurs where any sulfate containing source is incorporated into the concrete. Examples include the use of excess of added gypsum in the cement, sulfate-rich aggregate, or contamination. Proper testing and screening processes tend to avoid damage from internal sulfate attack.

##### ***2.1.2.1.1 Delayed Ettringite Formation***

Delayed ettringite formation (DEF) is a special case of internal sulfate attack. Delayed ettringite formation has been a significant problem in many countries. It occurs in concrete which has been cured at elevated temperatures, for example, where steam curing has been used. It was originally identified in steam-cured concrete railway

sleepers (railroad ties). It can also occur in large concrete pours where the heat of hydration has resulted in high temperatures within the concrete (Guide to Durable concrete, ACI, 2016). Ettringite forms when penetrating sulfates react with AFm ( $\text{Al}_2\text{O}_3\text{-Fe}_2\text{O}_3\text{-mono}$ ). However, recent investigations on the mechanisms of sulfate attack show that aluminum released from C-A-S-H phases in hardened pastes made with Portland cement/fly ash binder compositions also contributes to the formation of ettringite (Müllauer, Beddoe & Heinz, 2012). DEF causes expansion of the concrete due to ettringite formation within the paste and can cause serious damage to concrete structures. DEF is not usually due to excess sulfate in the cement, or from sources other than the cement in the concrete. Although excess sulfate in the cement would likely increase expansion due to DEF, it can occur at normal levels of cement sulfate (Skalny, Marchange & Odler, 2002). Important point in understanding DEF is that ettringite is destroyed when heat around  $70^\circ\text{C}$ . DEF occurs if the ettringite which normally forms during hydration is decomposed, then subsequently re-forms in the hardened concrete. In normal concrete, the total amount of ettringite which forms is evidently limited by the sulfate contributed by the cement initially. Damage to the concrete occurs when the ettringite crystals exert an expansive force within the concrete as they grow. It follows that the quantity of ettringite which forms is relatively small.

Ettringite crystals form widely dispersed throughout the paste. If expansion causes cracking, ettringite may subsequently form in the cracks but this does not mean the ettringite in the cracks caused the cracks initially (Skalny, Marchange & Odler, 2002). DEF causes a characteristic form of damage to the concrete. While the paste expands, the aggregate does not. Cracks form around these non-expanding 'islands' within the paste -

the bigger the aggregate, the bigger the gap. The cement paste has expanded, and a gap has formed between the aggregate and the cement paste. The aggregate is no longer contributing to concrete strength since it is effectively detached from the cement paste. Often, these gaps become filled with ettringite. In severe cases, the migration of sulfate ions can be accompanied by a gradual dissolution of  $\text{Ca}(\text{OH})_2$  and decomposition of the calcium silicate hydrate (C-S-H) phase. This continues if the pH is within a range where ettringite is stable (10.7–12.5), (Bentz, Ehlen, Ferraris, & Winpigler, 2002), (Ferraris, Stutzman, Peltz, & Winpigler, 2005). In laboratory tests, limestone coarse aggregate has been found to reduce expansion. DEF usually occurs in concrete which has either been steam cured, or which reached a high temperature during curing because of the exothermic reaction of cement hydration. As the curing temperature of concrete increases, ettringite normally persists up to about 70° C. Above this temperature it decomposes. In mature concrete, monosulfate is usually the main sulfate-containing hydrate phase and this persists up to about 100° C. DEF could occur in concrete, which was heated externally, e.g. from fire (Lawrence, 1993). The effect of cement composition on DEF may not be completely well understood. In some laboratory tests, DEF expansion was shown to correlate positively with cement-related factors, including high sulfate, high alkali, high MgO, cement fineness, high  $\text{C}_3\text{A}$ , high  $\text{C}_3\text{S}$ .

#### ***2.1.2.2 External sulfate attack***

External sulfate attack can generally be thought as when the source of sulfate is found outside the concrete matrix and penetrates into the cementitious systems usually through solution (i.e., pore water). In addition, the type of attack can also be

subcategorized into physical (e.g., salt crystallization) or chemical (e.g., reformation of cement hydration) damage on the cementitious matrix. This section describes mechanisms and published data related to the chemical form of external sulfate attack.

#### *2.1.2.2.1 Chemical external sulfate attack*

This is the more common type and typically occurs where water containing dissolved sulfate penetrates the concrete. A well-defined reaction front can often be seen in polished sections; ahead of the front the concrete is normal, or near normal. Behind the reaction front, the composition and microstructure of the concrete will have changed. These changes may vary in type or severity but commonly include extensive cracking, expansion, loss of bond between the cement paste and aggregate, alteration of paste composition, with monosulfate phase converting to ettringite and, in later stages, gypsum formation (Liu, Chen, Wang & Yu, 2020). The necessary additional calcium is often provided by the calcium hydroxide and calcium silicate hydrate in the cement paste.

The effect of these changes is an overall loss of concrete strength. The above effects are typical of attack by solutions of sodium sulfate or magnesium sulfate. Solutions containing magnesium sulfate are generally more aggressive, for the same concentration. This is because magnesium also takes part in the reactions, replacing calcium in the solid phases with the formation of brucite (magnesium hydroxide) and magnesium silicate hydrates. The displaced calcium precipitates mainly as gypsum. For example, attack from solutions containing  $\text{Na}_2\text{SO}_4$  as the primary source typically involves the formation of ettringite and gypsum leading to extensive cracking and volume changes. On the other hand, soils and seawater containing magnesium sulfate ( $\text{MgSO}_4$ )

can lead to the formation of other deleterious products, such as brucite. The magnesium cation also has a strong affinity to replace calcium in the C-S-H phase. This results in a gradual loss of its binding properties and thus, an apparent reduction in strength.  $\text{MgSO}_4$  has a far more damaging effect as compared with that of other sulfates because of its reaction with calcium silicates, in addition to the formation of gypsum and ettringite in the hardened paste system (Dhole, 2008), (Drimalas, 2007). Other commonly found sulfates in soils and groundwater include potassium and calcium sulfate, however, their lower solubility in solution is generally believed to result in a slower rate of degradation. Nonetheless, these sulfate types are commonly found in combinations in the field increasing the severity and complexity of the sulfate attack mechanism (Neville, 1995).

#### *2.1.2.2.2 Thaumasite*

The thaumasite form of sulfate attack (often abbreviated to TSA) requires a source of sulfate and a source of carbonate. Thaumasite can form in concrete and in mortar. The cement hydration products normally present, mainly calcium silicate hydrate and calcium hydroxide, are decomposed because of both sulfate attack and of carbonation. Since it is the calcium silicate hydrate in concrete that provides most of the strength, thaumasite formation results in severe weakening of the concrete microstructure. Thaumasite has the chemical formula:  $[\text{Ca}_3\text{Si}(\text{OH})_6 \cdot 12\text{H}_2\text{O}] (\text{SO}_4)(\text{CO}_3)$  or  $\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$  (Rahman & Bassuoni, 2014).

Thaumasite tends to form at low temperatures, typically about  $4^\circ\text{C}$  -  $10^\circ\text{C}$ ; however, studies have shown the formation of thaumasite at room temperature. In laboratory investigations it was shown that thaumasite forms more rapidly in a

temperature range below 15°C (ideally about 5°C) while ettringite prefers an environment of more than 15°C, however, both minerals are able to form under opposite conditions (Brueckner, Williamson & Clark, 2012). As it forms, the concrete or mortar converts to a friable material often described as a 'mush.' Concrete severely suffering from thaumasite formation can easily be broken with the fingers and therefore the coarse aggregate lifted out. A source of additional water is additionally required for thaumasite formation. Damp or wet cementitious render over brickwork, specially where the render might be cracked, and masonry and concrete in cool or damp cellars are samples where thaumasite might occur. Sulfate attack tends to result in the formation of ettringite. This uses aluminum provided by the cement and clearly this is often limited in quantity in normal concrete. However, aluminum is not involved in the formation of thaumasite; given an adequate supply of carbonate and sulfate, can still form thaumasite until the calcium silicate hydrate is decompose completely (Luo, Zhou, Wang & Fang, 2019). The formation conditions for TSA in above ground structures hardly differ from buried concretes apart from the situation of the sulfate, which is already available within the material of above ground constructions with the exception of ground floor slabs in touch with an external source of sulfate. Structural consequences of TSA in buried concrete structures can be the loss of strength due to reduction of cross-sectional area, possible premature corrosion due to loss of cover concrete, loss of sliding resistance towards lateral movement and reduction in skin friction. Actual cases of loss of structural integrity in the field have not been found but a structure in the Canadian Arctic, where the columns supporting a building had to be replaced after two years in aggressive environment (Bickley, Hemmings, Hooton & Balinsky, 1995), (Skalny & Thaulow,

2002). The determination of the rate of TSA deterioration is very complicated due to the many interrelated factors that affect it such as; the availability and concentration of both sulfate and carbonate ions dissolved in groundwater, the quality and type of concrete, and the range of temperature (Thaumasite Expert Group, 1999), (Crammond, 2003). The tensile strength is a very expressive value for the measurement of resistance against TSA because the generally used determination measurements are the expansion, the reduction in compressive strength and the loss of mass. Whereas for the visual form of estimation of damage due to TSA is a very pragmatic method of initial investigation in the field and it is not relevant for the description of thaumasite progress in laboratory conditions. TSA is expected to occur at the full sample area exposed to aggressive solution (Brueckner, Williamson & Clark, 2012).

### **2.1.3 Mitigating Sulfate Attack**

#### ***2.1.3.1 Prescriptive approach***

A prescriptive specification describes a product predominantly by its composition. For example, ASTM C 150 limits tricalcium aluminate ( $C_3A$ ) levels for Type II and V Portland cements of 8 % and 5 %, respectively, to control sulfate resistance. In new concrete structures, methods to mitigate or minimize damage from external sulfate attack have been primarily based on prescriptive requirements. One of the most common ways of protecting against sulfate attack is to reduce the alumina content by limiting the  $C_3A$  in Portland cement. Historically, Type II Portland cement (with  $C_3A$  between 5 and 8 percent) and Type V Portland cement (with  $C_3A$  less than 5 percent) have been specified for moderate and severe sulfate environments, respectively. The use of slag cement is

also an extremely effective way of reducing the potential for sulfate attack (Guide to Durable concrete, ACI, 1992); (Skalny, Marchange & Odler, 2002). The use of slag cement reduces the likelihood of sulfate attack in three ways. Firstly, slag cement does not contain  $C_3A$ , so its addition in concrete dilutes the total amount of  $C_3A$  in the system. Secondly, slag cement reduces concrete permeability, making it harder for sulfates to penetrate and permeate through the concrete matrix. Lastly, slag cement reacts with excess  $Ca(OH)_2$  to form additional calcium silicate hydrate gel (the “glue” that provides strength and holds the concrete together). This decreases the total amount of  $Ca(OH)_2$  in the system that may be available to react with external sources of sulfate.

Used in the proper proportions, slag cement can give a Type I cement the sulfate resisting properties of a Type II cement (usually 25 to 50 percent slag cement replacement for Portland), and it can give a Type I or a Type II cement the sulfate resisting properties of a Type V cement (usually 50 to 65 percent slag cement replacement for Portland) (Guo, Jin-Jun, Wang, Kun, Guo, Ting, Peng, 2019). For this combination of materials, 15 and 25 percent slag cement replacement achieved moderate sulfate resistance, and 35 and 50 percent achieved high sulfate resistance, based on ASTM C989 six-month expansion limits (ASTM C1012, 2001).

According to Biczok, the mechanism of sulfate attack can change with a change in the concentration of the sulfate solution. For attack by sodium sulfate solution, at a low concentration of sulfates ( $< 1000 \text{ ppm } SO_4^{2-}$ ), the primary product deposited is ettringite, while at high concentrations ( $> 8000 \text{ ppm } SO_4^{2-}$ ), gypsum is the main product. In the intermediate range ( $1000\text{--}8000 \text{ ppm } SO_4^{2-}$ ), both gypsum and ettringite are observed (Biczok, 1967).

Ping and Beaudoin proposed a new theory of sulfate-related expansion based on thermodynamics. According to them, ‘crystallization pressure’, which is a result of the interaction between attack products such as ettringite, and the cement paste, is responsible for expansion. Two conditions are necessary to maintain the crystallization pressure, namely, the growth of the crystal in a confined region, as well as a high concentration of reactants that drive the reaction. Using this philosophy, they suggest that the best way to minimize the expansion is by reducing the concentration of  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  in the solution. However, no experimental evidence was provided related to the effect of  $\text{SO}_4^{2-}$  concentration. The temperature of the solution was identified as an important factor controlling the expansion of the specimens. Expansion was determined to be proportional to the temperature of the solution. The principal effect of temperature, according to the theory proposed, was to increase the crystallization pressure due to the formation of the solid product (Ping & Beaudoin Part I & II, 1992).

A few researchers (ASTM C989-99, 2001), (Dhole, 2008), (Drimalas, 2007), (Neville, 1995) have indicated that the utilization of a suitable cement and supplementary cementitious materials (SCMs), as well as minimizing permeability using a low w/c, can demonstrate satisfactory performance for concrete exposed to sulfate-rich environments. The use of a sulfate-resistant Portland cement such as Type II or Type V can reduce the severity of attack by limiting the tricalcium aluminate ( $\text{C}_3\text{A}$ ) available to react and form secondary ettringite, which is the phase that causes expansions when formed in small pores within a certain size range (Stark, 1989). As such, so as to control the obstruction against sulfate attack, ASTM C150 constraints the  $\text{C}_3\text{A}$  content for sulfate resistant cements to 8% and 5% for Type II and Type V, respectively. Many accelerated test

methods are carried out with higher w/cm ratio and smaller test specimens so that the degradation process can be accelerated, one such study is the Ferraris et al (Ferraris, Stutzman, Peltz & Winpigler, 2005). This research studied that the specimen size also has an effect, showing that smaller size prisms (10 by 10 by 100 mm [0.4 by 0.4 by 1.5 in.]) increased the expansion rate, enhancing the test results in much less time than the standard 25 by 25 by 285 mm (1 by 1 by 11.25 in) specimens. Ferraris et al (Ferraris, Clifton, Stutzman & Garboczi, 1997). also tested cylinders with constant length of 152 mm (6 in.) and varying diameters (25, 50, 75 mm [1, 2, 3 in.]) exposed to sulfate solutions from the sides, concluding that the ionic diffusion mostly governs the expansion from external sulfate attack and hence, could be accelerated using smaller specimens. However, paste samples were used as test specimens, which do not reflect field conditions accurately.

Several researchers have shown that the use of appropriate combination of cement and supplementary cementitious materials (SCMs), as well as minimizing permeability using a low w/c, can demonstrate satisfactory performance for concrete exposed to sulfate-rich environments. The use of a sulfate-resistant portland cement such as Type II or Type V can reduce the severity of attack by limiting the tricalcium aluminate ( $C_3A$ ) available to react and form secondary ettringite, which is the phase that causes expansions when formed in small pores within a certain size range (Dhole, 2008), (Drimalas, 2007). To control the resistance against sulfate attack, ASTM C150, Standard Specification for Portland Cement, (Standard Specification for Portland Cement, 2018) limits the  $C_3A$  amount to 5 % and 8 % for Type V and Type II cements, respectively.

In the studies done by ..(Kurtis, Shomglin, Monteiro, Harvey & Roesler, 2001)., though hardened cement specimens suffered the formation of ettringite by sulfate attack can, however, does not generally, bring about expansion and lead to cracking of the cement. The ettringite-type of attack is by and large less in the field since there are several well-known ways to keep away from this type of attack (i.e., reducing the amount of  $C_3A$  and right proportioning of  $C_3A$  and gypsum in cement). Also, gypsum can be produced during sulfate attack through cation exchange reactions (Mehta & Monteiro, 1996). The arrangement of gypsum itself may result in just moderately limited amount of expansion, when compared to the ettringite type of sulfate attack. Response of calcium hydroxide to shape less-dissolvable items diminishes the pore solution alkalinity. At the point when calcium hydroxide is depleted, C-S-H, the essential quality providing strength to the hydrated cement paste, experience decalcification what is more, may get unstable. This kind of loss in adhesion and strength is indicative of a sulfate attack.

In commercial practice, the dosage of fly ash is limited to 15%-20% by mass of the total cementitious material. Usually, this amount has a beneficial effect on the workability and cost economy of concrete, but it may not be enough to sufficiently improve the durability to sulfate attack, alkali-silica expansion, and thermal cracking. For this purpose, larger amounts of fly ash, on the order of 25%-35% are being used. Although 25%-35% fly ash by mass of the cementitious material is considerably higher than 15%-20%, this is not high enough to classify the mixtures as HVFA concrete according to the definition proposed by Malhotra and Mehta. From theoretical considerations and practical experience, the authors have determined that, with 50% or more cement replacement by fly ash, it is possible to produce sustainable, high

performance concrete mixtures that show high workability, high ultimate strength, and high durability. (Malhotra & Mehta, 2002)

Note that the deterioration frequently reported in the field isn't brought about by ettringite formation but due to the deterioration of CH and C-S-H to gypsum by sulfate ions and transformation of these hydration items to aragonite, apparently because of carbonation (Mehta, 1992). Cracking caused by ettringite or gypsum formation and ettringite expansion as well as loss of C-S-H will adversely affect compressive strength. Thus, both forms of sulfate attack are considered when loss in strength is used as the measure of damage experienced during sulfate exposure (Kurtis, Shomglin, Monteiro, Harvey & Roesler, 2001).

#### ***2.1.3.2 Performance approach***

A performance specification describes how well a product must perform. The performance-based ASTM C 1157 utilizes a physical test (ASTM C 1012) for sulfate resistance by evaluating expansion of mortar prisms made with the cement and requiring them to have expansion below a certain limit without specifying the cement composition limits. Thus, critical issues about test strategies for evaluating sulfate resistance are constantly being concentrated by numerous researchers. Additionally, experiments are often carried out utilizing a mortar specimen or a small paste with a moderately high water-to-cementitious material proportion (w/cm) and immersed in an aggressive sulfate solution to accelerate the attack mechanism. The commonly used salts for sulfate obstruction tests are  $\text{Na}_2\text{SO}_4$  and  $\text{MgSO}_4$  because of their higher solvency and their forceful nature against cement hydrates.

While several researchers have commonly utilized a single sulfate solution at a single concentration level, distinctive sulfate solutions and concentrations as well as different physical parameters to qualify sulfate resistance of cementitious materials have additionally been explored. For instance, Koch (Köch & Steinegger, 1960) decided the sulfate obstruction of mortars by the decline in flexural strength between tests (samples) put in Na<sub>2</sub>SO<sub>4</sub> and similar tests (samples) put away in deionized water. Different tests, for example, the ASTM C1012 test method, (ASTM C1012/C1012M-18a, 2018) monitor the expansion of mortar bars placed in a 5 % Na<sub>2</sub>SO<sub>4</sub> solution and can satisfy the standard if the mortar bars observe an expansion  $\leq 0.10$  %. The testing solution is periodically replenished in the ASTM 1012 test whereas the other tests continue to use the same solution throughout the test. Mehta and Gjorv (Mehta & Gjorv, 1974) created a mechanism that circulates the solution in which the pH is kept steady by manual titration with H<sub>2</sub>SO<sub>4</sub>. Mehta (Mehta & Monteiro, 2006) also automated the above-mentioned strategy continuously titrating it with H<sub>2</sub>SO<sub>4</sub>, using pH controller that controls and monitors the pH of the solution. As indicated by Mehta, the system subtleties were sufficient to yield reproducible outcomes. Brown (Brown, 1981) utilized a similar test arrangement revealing that controlling the pH of the sulfate solution even more precisely represents the field conditions and prompts a quicker expansion of mortar bars than a typical pH sulfate solution that is not controlled.

Like the study done Mehta, the recent study done by Huang, Q explains that the sulfate attack on mortars was accelerated using electrical pulse as an external electrical field. Meanwhile, the specimens with the same mix proportions were immersed in sulfate solution as comparison. The sulfate concentration and mechanical properties, including

flexural and compressive strengths, were measured to evaluate the effects of sulfate attack. In addition, the transformation of microstructural nature and phase of the specimens were evaluated by scanning electron microscope and X-ray diffraction. The results showed that electrical pulse greatly accelerated the penetration of sulfate ions into the mortars and resulted in more ettringite and gypsum formed, leading to a greater depth of sulfate reaction. In the electrical pulse test, the sulfate content in  $MgSO_4$  solution was greater than that in  $Na_2SO_4$  solution, which was the opposite case in immersion test. Moreover, the electrical pulse resulted in significant mechanical strength losses in a much shorter period as compared with those in immersion test. These findings indicated that the electrical pulse accelerated sulfate attack could be a new acceleration test for evaluating the sulfate resistance of cement-based materials (Huang, Wang, Yang, Zhou & Yin, 2015).

More recently, the study conducted by Huang et al. used the techniques of introducing electrical field which accelerated the sulfate attack and induced deterioration, as it facilitated the ingress of sulfate ions into the matrix of mortar and the formation of expansive gypsum and ettringite (Huang, Wang, Zeng, Yang, Luo, & Yang, 2016). The results showed that the combination of sulfate attack and electrical field accelerated the damage process of mortars when compared with sulfate attack alone. Other researchers have proposed other innovative techniques to determine sulfate resistance namely Huang et al (Huang, Wang, Yang, Zhou & Yin, 2015). used an electrical pulse cycle as an external electrical field to accelerate the migration of sulfate into mortar bars and thus, accelerate the attack. They found that the electrical pulse resulted in significant mechanical strength loss because of the formation of massive sulfate products

According to Moir, increasing the w/c-ratio to 0.60, and thus increasing the porosity of the concrete, should shorten the time scale necessary to penetrate between cement pastes which are inherently sulfate resisting and those which are not (Moir, 1999). But the disadvantage of the use of high w/c-ratio is that a greater w/c-ratio creates more pores and thus more place for the reaction products, so expansion will occur later (Monteny, Vincke, Beeldens, De Belie, Taerwe, Van Gemert & Vertraete, 2000).

In the study done by Irassar a new criterion for evaluation of sulfate resistance was described, which he called the 'crack-time'. Flexural strength development was used as the test parameter to evaluate the different phases of sulfate attack including the following: filling of pores, cracking, strength loss, and deterioration of the structure. With time, the flexural strength of mortar bars in sulfate solution was found to increase up to a maximum, and then decrease rapidly (Irassar 1990). A relatively new procedure developed by Mulenga et al. focus on the analysis of the relative tensile strength of concrete prisms ( $40 \times 40 \times 160 \text{ mm}^3$ ) stored in sulfate solution in relation to water stored specimen. The prisms are therefore vacuum saturated by and immersed in sodium sulfate solution of  $33.9 \text{ g SO}_4^{2-}/\text{l}$  at  $8 \text{ }^\circ\text{C}$ . It is stated that the method allows to achieve repeatable results regarding the sulfate resistance of a concrete. However, the application of tensile strength on prisms as the basic measurement principle results in a relatively high effort for testing and a large scatter compared to other test methods (Mulenga, Zum Sulfatangriff auf Beton, 2002).

Heinz, D conducted a study in which a specially constructed stress cell was used to measure the stress generated in thin-walled mortar cylinders caused by exposure to  $\text{Na}_2\text{SO}_4$  solutions with sulfate concentrations of 1.5 and 30 g/L at different degrees of

restraint as well as the unrestrained free expansion. The mineralogical reactions were investigated by X-ray diffraction and changes in the pore size distribution measured by mercury intrusion porosimeter. It was thus possible to distinguish between the crystallization processes responsible for damage initiation and subsequent crystallization in the damaged matrix. The results can be understood in terms of the effect of crystal surface energy and size on supersaturation and on crystal growth pressure (Müllauer, Beddoe & Heinz, 2013).

#### **2.1.4 Field Performance**

In the recent studies, ASTM test methods for predicting sulfate resistance have been criticized for failing to adequately predict field performance. For which a recent investigation done by Kurtis, K. E, cement pastes were tested by an accelerated test method where pH and sulfate concentration remained constant, conditions that are more representative of field conditions. The results show that the pH-controlled accelerated test method used in this investigation yields data based upon measurements of compressive strength that are adequate to determine sulfate resistance of Portland and non-Portland cements (Kurtis, Shomglin, Monteiro, Harvey & Roesler, 2001).

In field conditions concrete is also subjected to atmospheric effects such as wetting and drying, which could accelerate the degradation procedure. Wetting/drying cycling, as used by Almeida and De Belie et al. could lead to extremely high sulfate concentrations and causes rapid deterioration due to sodium sulfate crystallization. Almeida found that, in the soaking and drying test, the porosity and the capillary absorption controlled the performance of concrete more than the chemical composition

(Almeida, 1991), (De Belie, 2007), (De Belie, Monteny, Beeldens, Vineke, Van Gemert & Vertraete, 2004).

### **2.1.5 Influence of Sulfate Attack**

A two-stage process is involved in the expansion of mortar specimens under sodium sulfate solution exposure. The expansion is very low during the initial stage. Followed by this stage a sudden increase in the expansion occurs where the rate of expansion is nearly constant until failure. In the study done by Manu Santhanam & Menashi D Cohen the effects of using various sulfate concentrations and of using different temperatures are also reported. The results suggest that the expansion of mortars in sodium sulfate solution follows a two-stage process. In the initial stage, Stage 1, there is little expansion. This is followed by a sudden and rapid increase in the expansion in Stage 2. Microstructural studies suggest that the onset of expansion in Stage 2 corresponds to the appearance of cracks in the chemically unaltered interior of the mortar. Beyond this point, the expansion proceeds at an almost constant rate until the complete deterioration of the mortar specimen (Santhanam, Cohen & Olek, 2002).

The severity of damage depends on the type and concentration associated with the sulfate solution the concrete structure is subjected to, which will lead to cracking, volume changes, scaling and gradual loss in strength (Skalny, Marchange & Odler, 2002). For example, when solutions containing  $\text{Na}_2\text{SO}_4$  attack the structure they tend to form ettringite and gypsum which eventually leads to volume changes and extensive cracking. Whereas if the soil or seawater contains magnesium sulfate ( $\text{MgSO}_4$ ) it can lead to the formation of other injurious products, such as brucite. The magnesium cation also has a

strong affinity to replace calcium in the C-S-H phase which will eventually result in a gradual loss of its binding properties thereby affecting the strength.  $\text{MgSO}_4$  tends to have adverse effect when compared with other sulfates because of its reaction with calcium silicates and also, additionally forming ettringite and gypsum in the hardened paste system (ASTM C1012/C1012M-18a, 2018), (mb, Thomas, Folliard & Drimalas, 2013). Other commonly found sulfates in soils and groundwater include potassium and calcium sulfate, however, their lower solubility in solution is generally believed to result in a slower rate of degradation. Nonetheless, these sulfate types are commonly found in combinations in the field which tends to increase the complexity and severity of the sulfate attack mechanism.

Loss in strength due to the formation of ettringite and gypsum on the surface of concrete is the main threat to concrete structures in the field when exposed to sulfate attack. In the presence of a carbon dioxide source, e.g. limestone, at low temperatures, the precipitation of thaumasite also results in a severe loss in strength

Mass loss can also be used to determine the degradation rate. In the study done by Al-Amoudi, it is stated that for different cement blends, stored in mixed magnesium sulfate or sodium sulfate solutions, the deterioration is best assessed by the weight loss rather than length change. However, while using this parameter there is a need to be aware that it can lead to an increase in mass due to the formation of reaction products (Al-Amoudi, 1995).

### 3. METHODOLOGY

#### 3.1 Materials and Mix Proportion

##### 3.1.1 Fly ashes

The use of acceptable SCMs can also improve the resistance of concrete to sulfate attack by significantly reducing the ingress and movement of external sulfate ions into the concrete (Guide to Durable concrete, ACI, 2016). While the use of Class F fly ash typically improves sulfate resistance (mb, Thomas, Folliard & Drimalas, 2013), Class C fly ashes can impart inferior sulfate resistance as a result of the presence of reactive calcium-aluminate glass and crystalline phases (Drimalas, 2007), (mb, Thomas, Folliard & Drimalas, 2013) Class F fly ash is Pozzolanic in nature and in this test Type I & Type II cements are used with 25% and 30% replacement by mass of cement of Class C and Class F fly ash, respectively. Class F has low CaO typically less than 20% from burning anthracite or hard bituminous coals while Class C fly ash is pozzolanic with some hydraulic behavior. Also, Class C will typically have a high CaO content at 20% to 30% from burning softer lignite or subbituminous coals. In this study, both Class C and Class F fly ashes were used. By using different fly ashes, a variation in chemistry between the fly ashes is achieved so that a wider spectrum of performance can be achieved. Thereby providing enough variation to see if the test method performs well and helps identify the significance of each method.

Table 1 indicates the use of two different types of Class C (CA<sub>1</sub>, CA<sub>2</sub>) and Class F (FA<sub>1</sub>, FA<sub>2</sub>) fly ashes to provide a variation in the chemistry of the mixes.

### 3.1.2 Cement & Sand

Three types of ASTM C150 cements are used in the test namely type I cement (PC<sub>1</sub>) which is considered to be non-sulfate resistant whereas, type II (PC<sub>2</sub>) moderate sulfate resistant cement, and a high sulfate resistance type V cement (PC<sub>5</sub>). These cements were designated as the controls and were assumed to have low (poor), moderate, and high-sulfate resistance to external sulfate attack based on their C<sub>3</sub>A content as prescribed in ASTM C150. Several mixtures were cast in this study that included plain and blended cement with a high-calcium (CA) and low-calcium (FA) fly ash at 30 % and 25 % replacement by mass of cement, respectively. Table 1 provides the mixture proportions for the mortars investigated in this study.

All mortar mixtures were proportioned and prepared following the procedures prescribed in ASTM C109 (Using 2-in. Cube Specimen) (Liu, Chen, Wang & Yu, 2020), with Ottawa sand (ASTM C778) to cementitious (cement + SCM) ratio of 2.75; however, mixtures in this study was designed to have a constant w/cm ratio of 0.485 for all plain and blended Portland cement mixtures. Each mixture included an enough mortar cubes (50 mm) for determining compressive strength, and mortar prisms with a gauge length of 250 mm for measuring expansion and mass change before and after exposure to sulfate attack.

Table 1. Mixture proportions for mortar mixtures

ASTM C150 Portland Cements		Replacement Level of SCM (% cement by mass)			
		Class C (CA)		Class F (FA)	
Cement Type	W/CM	CA1	CA2	FA1	FA2
PC1	0.485	...	...	...	...
		30	...	...	...
		...	...	25	...
		...	30	...	...
PC2	0.485	...	...	...	...
		...	30	...	...
		...	...	...	25
PC5	0.485	...	...	...	...

Table 2. Sample mix ratio

Materials	Ratio (Mass)	Mass (needed)	Fly Ash Replacement (30%)	Cement (70%)
Cement	1.000	5109.38 g	1532.81 g	3576.56 g
Water	0.485	2478.05 g	-	
Sand	2.750	14050.78 g	-	
Total	-	21.64 kg	-	

The table 2 indicates the sample mix ratio used in the study, in which a 30% replacement of fly ash has been used and the water to cement ratio is 0.485 across all the tests. The fly ash replacement has varied to be either 25% or 30% with 2 different fly ashes under Class C and Class F, respectively.

Table 3. Nomenclature

Mix no.	Mix Name	Abbreviation
Mix 1	PC <sub>1</sub>	Type I cement
Mix 2	PC <sub>2</sub>	Type II cement
Mix 3	PC <sub>5</sub>	Type V cement
Mix 4	PC <sub>2</sub> (30% CA <sub>2</sub> )	Type II cement with 30 % Class C2 fly ash
Mix 5	PC <sub>1</sub> (30% CA <sub>1</sub> )	Type I cement with 30% Class C1 fly ash
Mix 6	PC <sub>1</sub> (30% CA <sub>2</sub> )	Type I cement with 30% Class C2 fly ash
Mix 7	PC <sub>2</sub> (25% FA <sub>2</sub> )	Type II cement with 25% Class F2 fly ash
Mix 8	PC <sub>1</sub> (25% FA <sub>1</sub> )	Type II cement with 25% Class F1 fly ash

Table 3 represents the nomenclature for the 8 different mixes used in the research.

It classifies the different types of cements used and the various percentage of fly ash combinations that were tested in this study. This table helps to provide an easy understanding for the terms used across the paper.

### **3.1.3 Apparatus**

All mortar mixtures were proportioned and prepared following the procedures prescribed in ASTM C109, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens) (ASTM C109/C109M-16a, 2018). The mortar prisms (25 by 25 by 285 mm) with a gauge length of 250 mm for measuring expansion. Mixer, Bowl and Paddle, an electrically driven mechanical mixer of the type equipped with paddle and mixing bowl is used for making the mixes. Also the hydraulic compression testing machine was used to measure the compressive strengths of the mortar cubes.

### **3.2 Testing Procedure**

The three following methods of testing follow the same initial procedures therefore, for all the three tests the specimens were batched and cast at the same time, also the specimens were cured for the same amount of time, and we demolded the same way. The exception would be for the pH-controlled testing method, due to the issues that occurred with the pH tank we had to redo a set of mixes separately for that test alone.

Therefore, for each batch of the mixes there were a total of 18 mortar prisms and 42 mortar cubes that were cast into the molds and heat cured in a sealed container above water while in oven at 50°C for 24 hours; Post this the prisms and cubes were all stripped

from their molds and subsequently transferred to a limewater bath for 6 additional days at 50°C until a curing age of 7 days was achieved. Thereafter, all the mixtures were evaluated for their compressive strength as a control at 7 days.

### **3.3 Controlled Method/Standardized Method (ASTM C1012)**

For the controlled ASTM 1012 method, once the 7-day curing was achieved they are taken out of the moist cabinet. For each batch of mix, a set of six mortar prisms and 18 mortar cubes were taken and subjected to the following procedure: firstly, each mortar prisms set were wiped down and immediately measured for their initial length and mass; thereafter, a set of four (4) mortar cube specimens were immediately tested for their initial 7-day strength using the compressive strength machine; lastly, all remaining 14 cube specimens along with the 6 mortar prisms were immediately submerged in an airtight storage container with 5% sodium sulfate solution at lab temperature (20°C). The mortar bars were continuously monitored for their length change in a periodical interval of say 1, 2, 3, 4, 8, 12, & 15 Weeks, and 4, 6, 9, 12, 15, & 18 months. Also, the remaining cubes were broken, and the compressive strength measured in an interval of 8, 12 & 15 weeks, unless failure occurred first.

### **3.4 Vacuum Impregnated Method**

Alike the controlled ASTM 1012 method, even the vacuum impregnated method will follow similar procedure in the beginning of the test and will vary at the end. For each batch of mix, a set of 6 mortar prisms and 14 mortar cubes were taken and subjected to the following procedure: firstly, each mortar prisms set were wiped down and immediately measured for their initial length and mass; thereafter, immediately the

mortar bars and the mortar cubes will be placed in oven at 38°C for a period of 14 days and air dried to remove moisture from the specimen to allow ingress of sulfate ions. After 14 days are over the mortar bars and cubes are removed from the oven and the initial length and mass of the bars and cubes are measured. The purpose of the drying period was to evaporate the pore water and permit the now-empty pores to be occupied by the sulfate solution (Aguayo, Drimalas & Folliard, 2019). Once the measurements are noted the mortar bars are placed vertically in the chamber along with the cubes and sealed in a transparent acrylic vacuum chamber. After sealing the specimen's, vacuum is introduced to the chamber for a period of 4 hours. Once the time is up we continue to maintain the vacuum in the chamber and simultaneously introduce 5% of sodium sulfate solution to the chamber and allow the mortar bars and mortar cubes to be exposed to vacuum and sodium sulfate solution for a period of 20 hours. Finally, the specimens are removed from the chambers and measure the specimen for the post vacuum change in expansion and mass of the specimens and leave it in an air tight container for static submersion in sulfate solution in the lab temperature (20°C). Similar to the ASTM 1012 test the mortar bars will be continuously monitored for their length change in a periodical interval (1, 2, 3, 4, 8, 12, & 15 weeks, and 4, 6, 9, 12, 15, & 18 months). Also, the remaining cubes will be broken, and the compressive strength will be tested in an interval of 8, 12 & 15 weeks.

### **3.5 pH-Controlled Method**

The pH-Controlled method will be an extension of the vacuum impregnation test. After the specimens are removed from the chamber, each are placed in a tank filled with sodium sulfate solution which has an automated titrating system which uses a 2.0

normality sulfuric acid which is pumped into the tank at a specific interval to maintain a pH of  $7 \pm 1$  throughout the entire test. Similar to the above two tests the mortar bars will be continuously monitored for their length change in a periodical interval (1, 2, 3, 4, 8, 12, & 15 Weeks, and 4, 6, 9, 12, 15, & 18 months). Also, the remaining cubes will be broken, and the compressive strength will be tested in an interval of 8, 12 & 15 weeks. Also, to maintain the pH of the tank the sodium sulfate solution needs to be replenished anytime between 3-4 weeks interval.



(A)



(B)



(C)

Figure 1. Test setup (A) ASTM C1012 (B) Vacuum impregnation (C) pH-Controlled

## 4. LENGTH CHANGE OF HYDRAULIC CEMENT MORTARS

### 4.1 Expansion in Controlled Method (ASTM 1012)

Observations of sulfate attack have proved that deteriorated concrete has undergone significant expansion. Internal stresses increase due to exposing the mortars to a 5% sodium sulfate solution and leading the mortar bars to swell and then crack. To calculate the expansion of the mortar bars (% length change) we use the formula,

$$\Delta L = \frac{L_x - L_i}{L_g} \times 100$$

where:  $\Delta L$  = Change in length at x age, %

$L_x$  = Comparator of specimen at x age-reference bar comparator reading at x age, inches/mm

$L_i$  = Initial comparator reading of specimen-reference bar comparator reading at the same time, inches/mm

$L_g$  = nominal gauge length, or 10 inches [250 mm] as applicable

The figure 2 explains the overall length change of hydraulic cement mortars across 8 different mixes occurring in the ASTM 1012 method. The major axis contains the number of exposure days i.e., the number of days submerged in the 5% sodium sulfate solution. The minor axis indicates the percentage of length change of the mortar bars. The gray dotted line indicates the 0.1% expansion of the mortar bars ( $\geq 0.10$  % expansion or fracture). Looking into this chart the PC<sub>1</sub> mortar bars and the PC<sub>1</sub> (30% CA<sub>1</sub>) mortar bars have completely broken right at 4 months and 6 months, respectively.

Whereas the PC<sub>2</sub> mortar bars and PC<sub>2</sub> (30% CA<sub>2</sub>) mortar bars have been broken at 12 months and 9 months, respectively. PC<sub>5</sub> and the rest of the three mixes seem to still do fine and the measurements are continued periodically.

Looking at the expansion rate of the PC<sub>1</sub> (30% CA<sub>2</sub>) and the PC<sub>2</sub> (30% CA<sub>1</sub>) have reached the 0.1% expansion almost around the same time (125 days). PC<sub>1</sub> seems to be the quickest to expand within 90 days (3 months). The PC<sub>2</sub>(25% FA<sub>2</sub>) has lasted longer and the >0.1% expansion has not occurred yet.

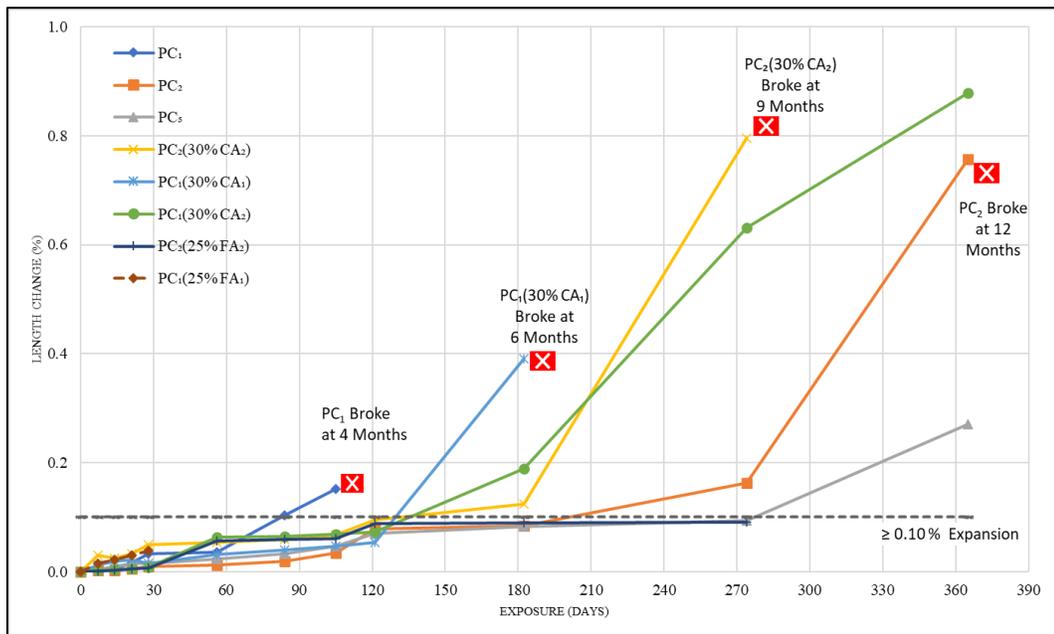


Figure 2. Expansion in Controlled Method (ASTM 1012) across 8 mixes

#### 4.2 Expansion in Vacuum impregnation Method

Looking into the graph in figure 3, the PC<sub>1</sub> mortar bars broke at 15<sup>th</sup> week, PC<sub>2</sub> mortar bars broke at the 9<sup>th</sup> month and the PC<sub>2</sub> (30% CA<sub>2</sub>) broke at the 4<sup>th</sup> month. Like the ASTM 1012 test method the PC<sub>5</sub> mortars bars are in a good shape and the rest of the

mixes are still being measured. PC<sub>1</sub> (Type I) 0.1% expansion had occurred around 40 days. Whereas looking into the C ashes PC<sub>1</sub> (30% CA<sub>2</sub>) and the PC<sub>2</sub> (30 % CA<sub>2</sub>) have expanded around the same time 120 days but the PC<sub>2</sub> (30 % CA<sub>2</sub>) did not last much long. The PC<sub>5</sub> and the PC<sub>2</sub> (25% FA<sub>1</sub>) have lasts much longer and the 0.1% expansion was around 120 days (4months) and 180 days (6months), respectively.

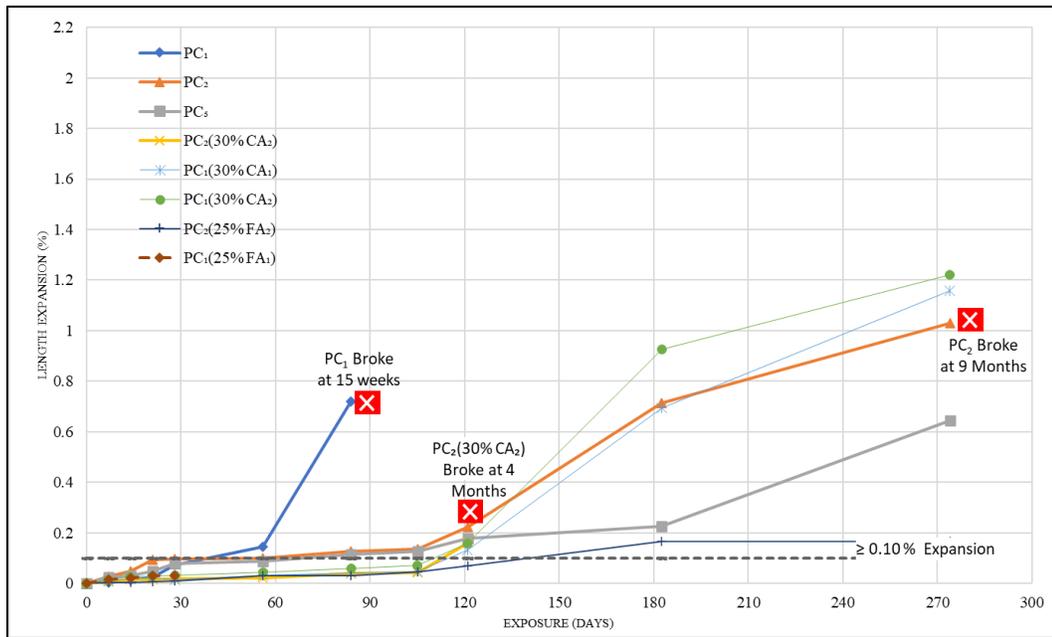


Figure 3. Expansion in Vacuum Method across 8 mixes

### 4.3 Expansion in pH-Controlled Method

Looking into the graph of figure 4, the PC<sub>1</sub> mortar bars broke at 12<sup>th</sup> week, PC<sub>2</sub> (30% CA<sub>2</sub>) broke at the 15<sup>th</sup> week and PC<sub>1</sub> (25% FA<sub>1</sub>) broke at the 12<sup>th</sup> week. Like the above test methods PC<sub>5</sub> mortars bars are in a good shape and the rest of the mixes are still being measured. The PC<sub>5</sub> bars crossed the 0.1% expansion rate around 2 months. Whereas all the rest of the bars reached the 0.1% expansion so quickly around a month (30 days). As expected the worst performing was the PC<sub>1</sub> (Type I) mix but it was

surprising to see PC<sub>1</sub> (25% FA<sub>1</sub>) to have reached the 0.1% expansion as quickly as two weeks. The PC<sub>1</sub> (30% CA<sub>2</sub>), PC<sub>1</sub> (30% CA<sub>1</sub>) and PC<sub>2</sub> (25% FA<sub>2</sub>) have all shown similar trend and exceeded the 0.1% expansion within 3 weeks. The expansion seems too aggressive and quick, also the graph depicts a sudden shoot up in values due to the pH-controlled sulfate environment.

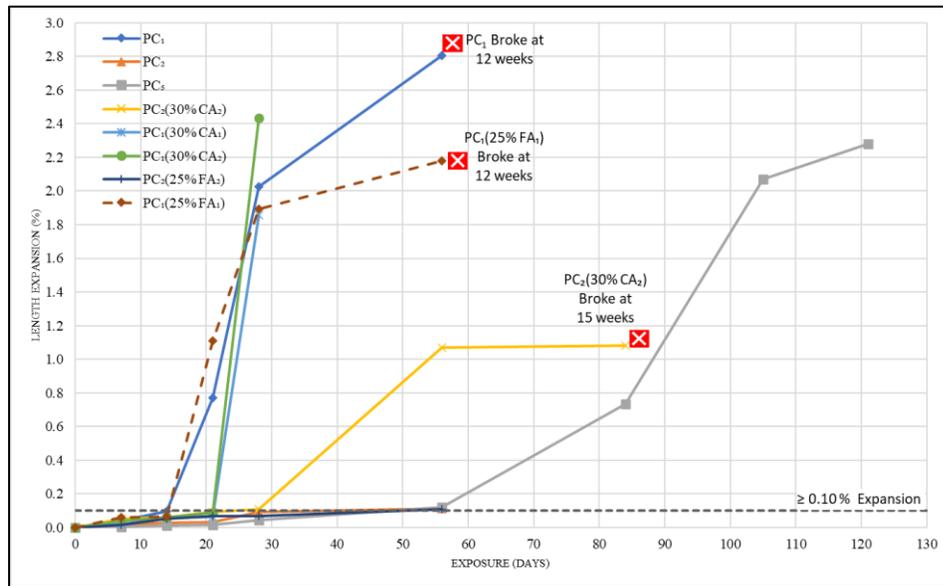


Figure 4. Expansion in pH Controlled Method across 8 mixes

## 5. COMPRESSIVE STRENGTH OF HYDRAULIC CEMENT MORTARS

### 5.1 Compressive strength change of hydraulic cement mortars across all the three test methods

This test method covers determination of the compressive strength of hydraulic cement mortars, using 2-in. or [50-mm] cube specimens. This test method provides a means of determining the compressive strength of hydraulic cement and other mortars. Using the compressive strength machine, a load of 200 to 400 lbs/s [900 to 1800 N/s] was applied on the cube specimen. To calculate the compressive strength of the cube the formula used is as follows:

$$f_m = P/A$$

where:  $f_m$  = compressive strength in psi,

P = total maximum load in lbs, and

A = area of loaded surface in (Guide to Durable concrete, ACI, 2016).

Compressive strength of the mortar cubes is tested after curing (prior to sulfate exposure) and after sulfate exposure. Loss in compressive strength resulting from sulfate exposure is used as a measure of the susceptibility of the cement to sulfate attack. A material will be considered sulfate susceptible if the average reduction in cube strength is greater than 25 percent.

The formula used to measure the average percentage of compressive strength change (loss/gain) is as follows,

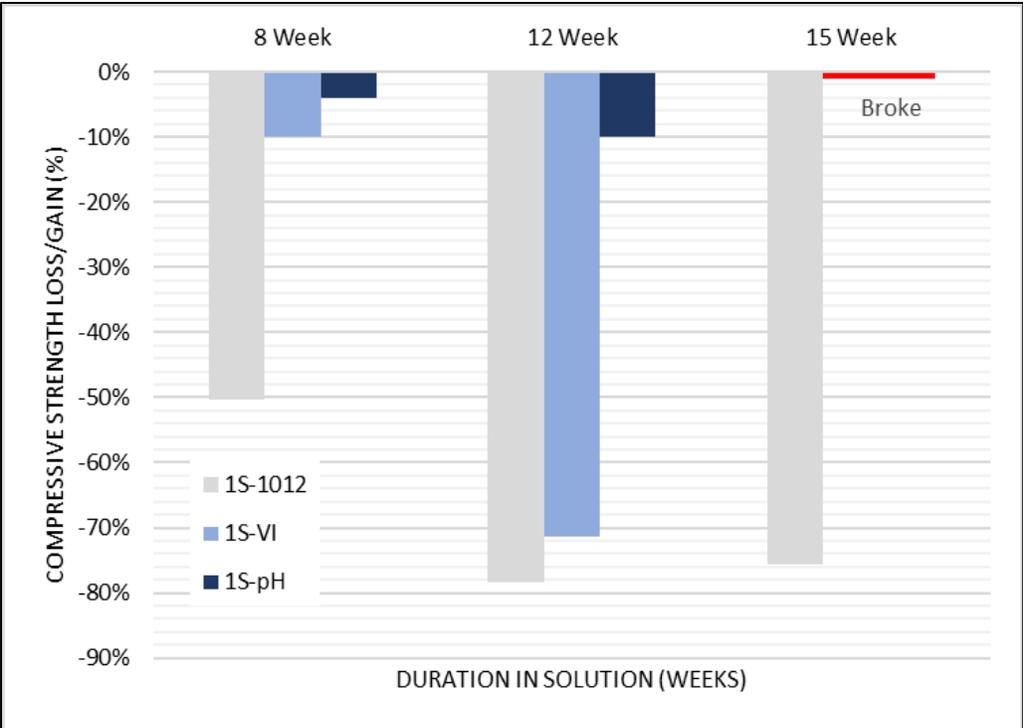
$$\Delta f_c = \frac{\Delta f_x - \Delta f_y}{\Delta f_x} \times 100$$

where:  $\Delta f_c$  = Average compressive strength change of the mortar cubes, %

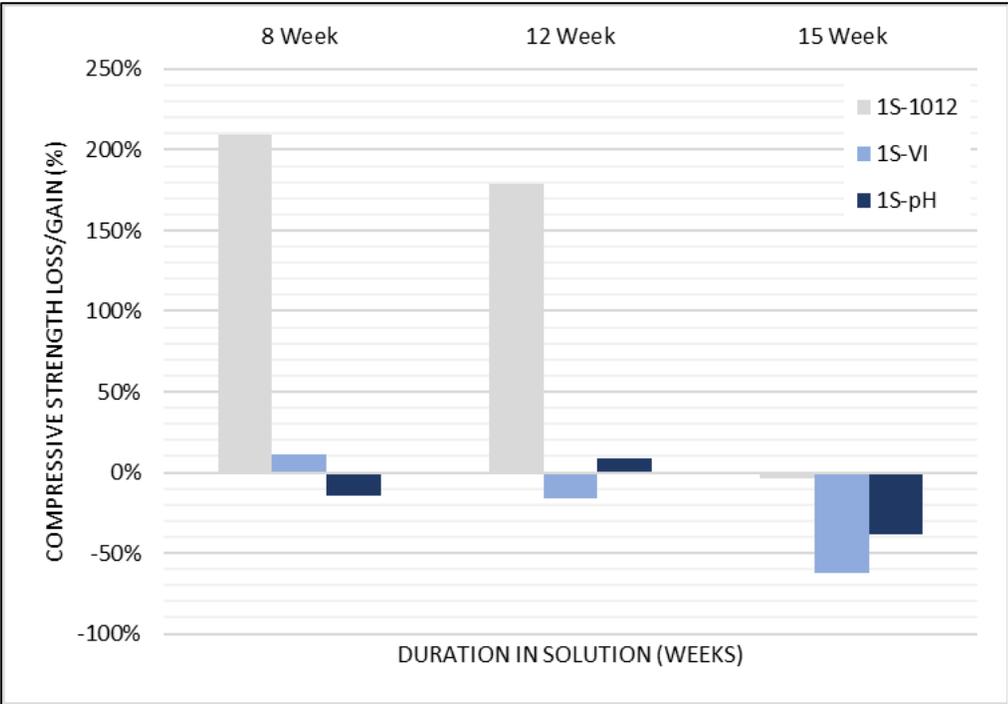
$\Delta f_x$  = Sum of the x day strength and x here indicates the initial 7-day strength, psi

$\Delta f_y$  = Sum of y day strength of the cubes and y can be the interval at which the cubes were measured, in this case 8, 12 & 15<sup>th</sup> week measurements, psi

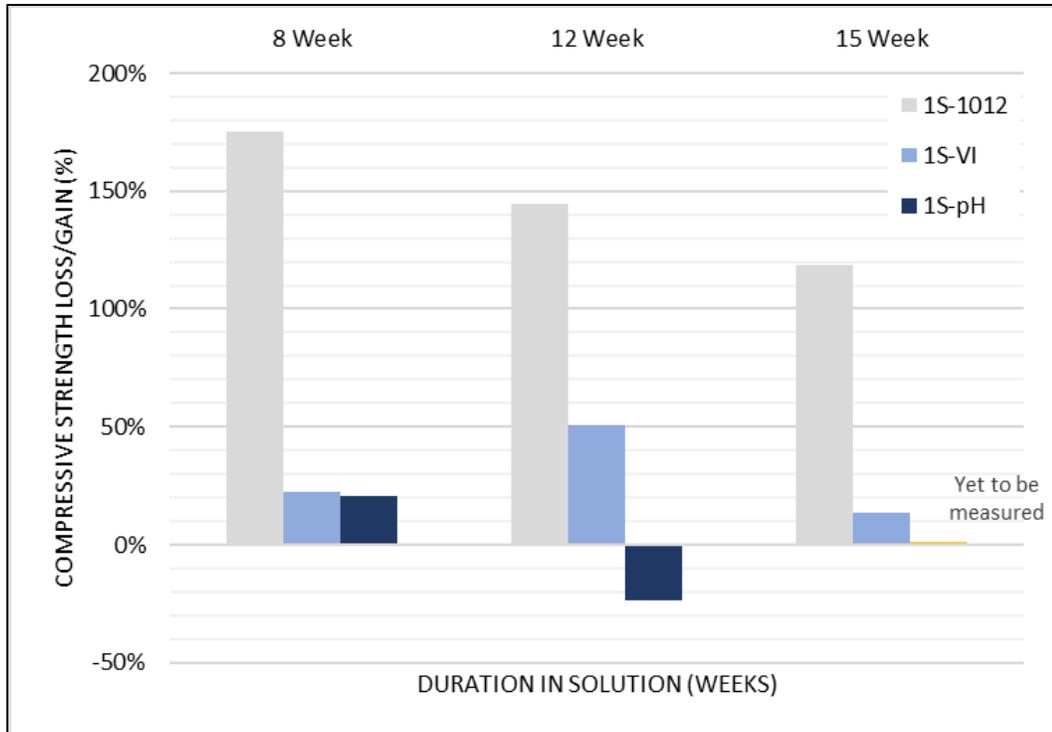
Figure 5(A). clearly states the comparison of the compressive strength across all the three tests of PC<sub>1</sub> mix (Type I cement) in which the mortar cubes of the Vacuum Impregnation and pH-controlled method have been completely broken even before attaining the 15<sup>th</sup> week measurement. Figure 5(B) indicates the compressive strength across all the three tests of PC<sub>2</sub> mix (Type I/II cement) in which we can see that it has performed better when compared to the PC<sub>1</sub> cubes. During the 8<sup>th</sup> and 12<sup>th</sup> week both the ASTM C 1012 and vacuum method have gained strength and deteriorated gradually. Whereas by the 15<sup>th</sup> week all the three methods have completely lost their strength on an average of close to 40% to 45%.



(A)



(B)



(C)

Figure 5. Comparing the compressive strength change of the PC<sub>1</sub>, PC<sub>2</sub> & PC<sub>5</sub> mix respectively where, (A) depicts the strength loss of PC<sub>1</sub> mix (B) depicts the strength loss/gain of PC<sub>2</sub> mix and (C) depicts the strength loss/gain of PC<sub>5</sub>.

The figure 5 (C) depicts the compressive strength loss/gain across all the three tests of PC<sub>5</sub> mix (Type V cement) from which we can clearly see that PC<sub>5</sub> has performed better when compared to the other two (no SCM cements) since its considered the best performing cement against sulfate attack. Even though the cubes have performed pretty well in the control and vacuum method the strength of the cubes have been considerably lost when it comes to the pH- controlled method. By 12<sup>th</sup> week we can see that the cube has lost strength close to 25% and looking at the remaining cubes they seem to have lost even more strength even though its yet to be measured.

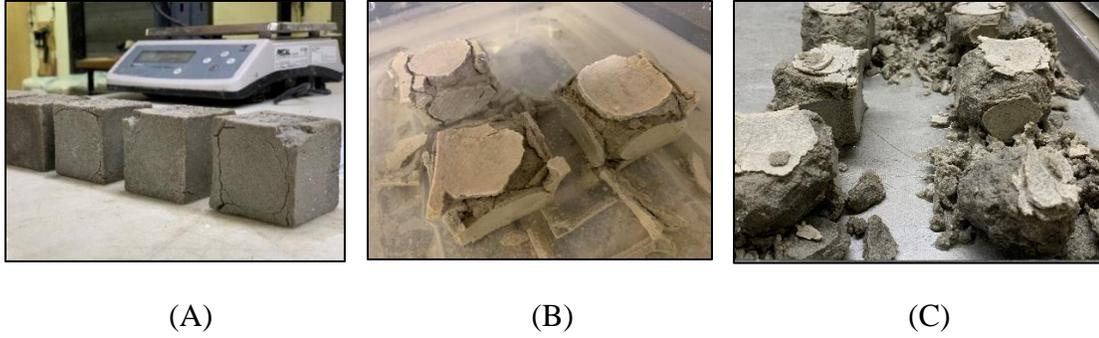


Figure 6. Visual deterioration of the PC<sub>1</sub> mix where, (A) depicts the strength loss of PC<sub>1</sub> mix at 8<sup>th</sup> week - Controlled - ASTM 1012 (B) depicts the strength loss of PC<sub>1</sub> mix at 12<sup>th</sup> week - Vacuum impregnation and (C) depicts the strength loss of PC<sub>1</sub> mix post 15<sup>th</sup> week - pH-Controlled.

We can infer from the visual deterioration in the figure 6. which clearly shows the difference in level of severity and damage from the use of the vacuum saturation procedure and pH-controlled method to accelerate the damage from sulfate attack. Thus, from the figures we can see that figure 6.(C) depicts the post 15<sup>th</sup> week strength loss of PC<sub>1</sub> (Type I) mix where the cubes completely were deteriorated and could not be measured anymore.

## 6. RESULTS AND DISCUSSION

### 6.1 Discussion on the length change of PC<sub>1</sub> with and without fly ashes across all the three tests

Figures 7,8 and 9 present the expansion results for all the plain PC<sub>1</sub> (Type I cement) and the combination of PC<sub>1</sub> (Type I cement) with class C and class F fly ashes were tested according to the controlled ASTM C1012 method, more accelerated vacuum impregnation method and the most accelerated pH-controlled method while placed in 5% Na<sub>2</sub>SO<sub>4</sub> solution (33,800 ppm SO<sub>4</sub><sup>2-</sup>). When compared with the ASTM C1012 method, the mortar bars showed a significant increase in the rate of expansion when subjected to the vacuum saturation procedure and even higher rate of expansion for the pH-controlled method. On an overall look on all the three figures its evident that the pH-controlled method is the most accelerated method with values spiking and PC<sub>1</sub> (Type I) cement being the lowest in sulfate resistance has failed real quick across ASTM C1012, vacuum impregnation and pH-controlled test attaining 0.1% expansion by 3 months, 8 weeks and 3 weeks, respectively. While looking into the two different class C ashes used we can infer that they have performed very similar to each other across the standard controlled test, vacuum impregnation, pH-controlled test and have reached the 0.1% expansion at 4 months, 12 weeks and 3 weeks, respectively. Mixtures in combination with 30 % CA behaved as expected based on previous testing and published data. (Dhole, 2008), (Drimalas, 2007).

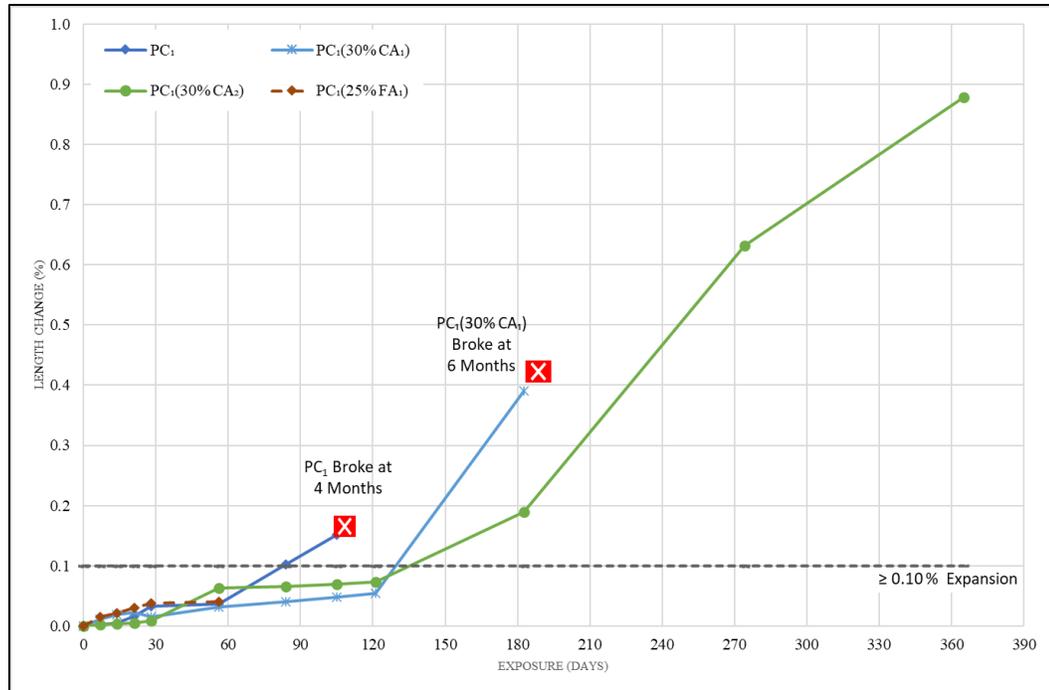


Figure 7. Expansion of the PC<sub>1</sub> based cement for the controlled ASTM C1012 test

On the other hand, class F ash has not yet reached the 0.1% expansion in the ASTM C1012 test method and the vacuum impregnation method since the measurements are still in progress and only 8 weeks of data has been recorded. From the chart in figure 7,8 and 9 and as mentioned earlier in the paper, it should be noted that mixture PC<sub>1</sub> (25% FA<sub>1</sub>) has only 8 weeks of data. This is because the mixture had to be re-done because at one point during the pH-controlled method, the specimens experienced acid attack. Rightly during that time, the construction work in the lab began and there were several hindrances in completing the test earlier thereby only few data points were collected unlike the other mixes. The class F ash has not yet reached the 0.1% expansion in the ASTM C1012 test method and the vacuum impregnation method since the measurements are still in progress and only 8 weeks of data has been recorded.

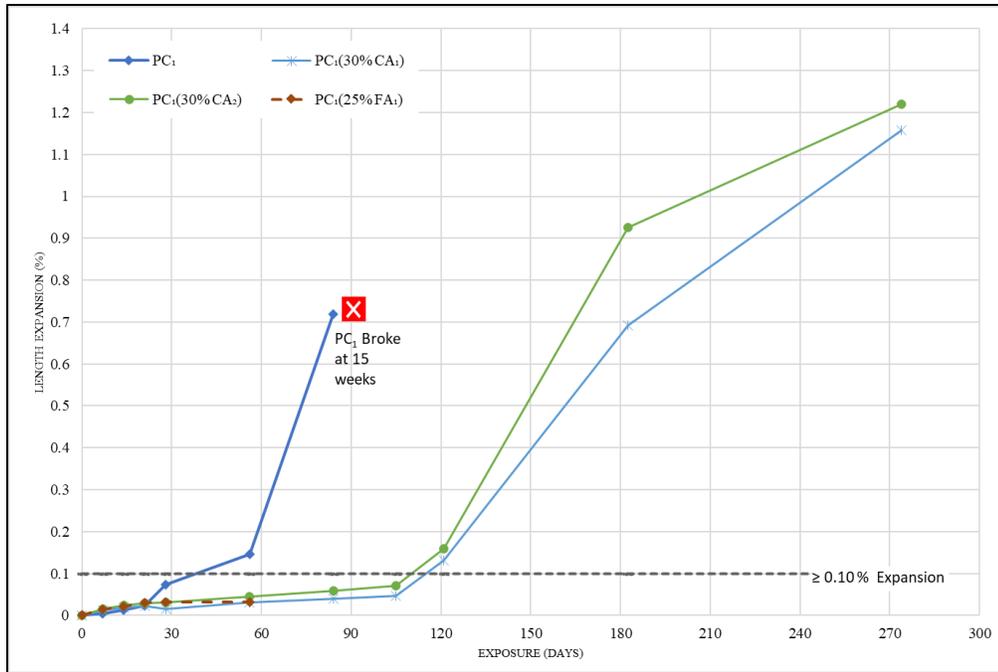


Figure 8. Expansion of the PC<sub>1</sub> based cement for the vacuum impregnation test

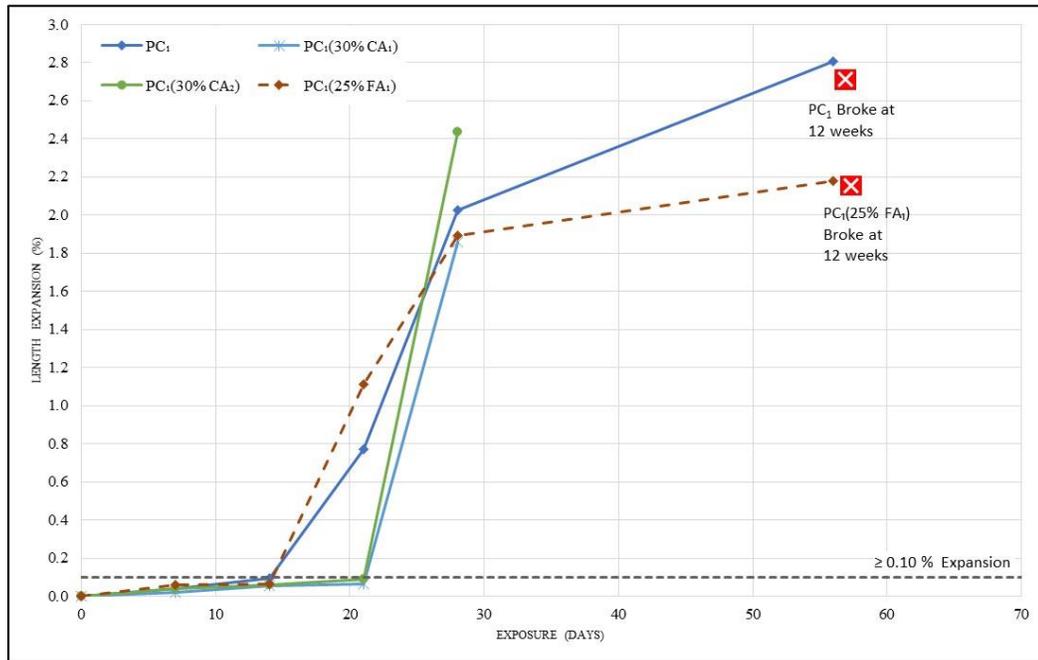


Figure 9. Expansion of the PC<sub>1</sub> based cement for the pH-controlled test

However, in the most accelerated pH-controlled test method the PC<sub>1</sub> 25% F ash mix has already exceeded the 0.1% expansion within 2-3 weeks and the bars were completely broke by the 12<sup>th</sup> week. When such sharp expansion is observed it is ideal to probably consider doing measurements more frequently to get more accurate measurements. Mixtures incorporating 25% FA exhibited the best performance to sulfate attack, as well as provided the most significant differences in expansion and time to failure between ASTM C1012, vacuum method and the accelerated pH-controlled method.

## **6.2 Discussion on the length change of PC<sub>2</sub> & PC<sub>5</sub> with and without fly ashes across all the three tests**

Figures 10, 11 and 12 present the expansion results for all the plain PC<sub>2</sub> (Type I/II cement) and the combination of class C and class F fly ashes along with PC<sub>5</sub> (Type V cement) tested according to the controlled ASTM C1012 method, more accelerated vacuum impregnation method and the most accelerated pH-controlled method while placed in 5% Na<sub>2</sub>SO<sub>4</sub> solution (33,800 ppm SO<sub>4</sub><sup>2-</sup>). The figure 10 indicates that in controlled ASTM C1012 method the PC<sub>5</sub> mixture performed too well with its 0.1% expansion occurring only around 9 months, whereas for the PC<sub>2</sub> the failure has occurred around 7 months. While comparing the PC<sub>2</sub> with 30% class C fly ash and 25% class F fly ash, the 25% F ash seems to have performed well and not exceeded the 0.1% expansion even past 9 months. Figure 11 also indicates that the class F fly ash performs better when compared to the class C ash which has exceeded the 0.1% failure rate around 12 weeks, but F ash sustains longer close to 5 months even under vacuum testing. Also, the PC<sub>2</sub> 30% C fly ash bars have completely broke around 4 months, but the PC<sub>2</sub> 25% F fly ash

has continued to last even past 9 months with slight cracks and spalling. Mixtures incorporating 25% F ash exhibited the best performance to sulfate attack, as well as provided the most significant differences in expansion and time to failure between ASTM C1012 and the vacuum method. Whereas, the chart in figure 12 indicates the most accelerated test method where the pH was controlled and in this case both the PC<sub>2</sub> with class C & F ashes reached the 0.1% expansion level around 4 weeks and 8 weeks, respectively. This indicates that the specimens that were vacuum impregnated and had the pH controlled showed highly accelerated sulfate attack, within 8 weeks results were achieved. Thereby, the pH-controlled method becomes the most accelerated method and even the best performing F ash had suffered aggressive sulfate attack.

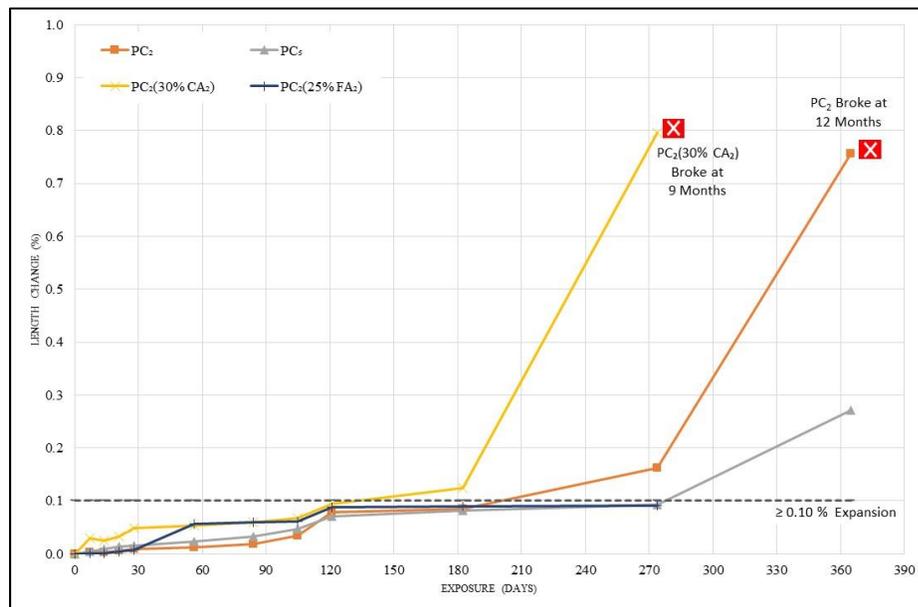


Figure 10. Expansion of the PC<sub>2</sub> & PC<sub>5</sub> based cement for the controlled ASTM C1012 test

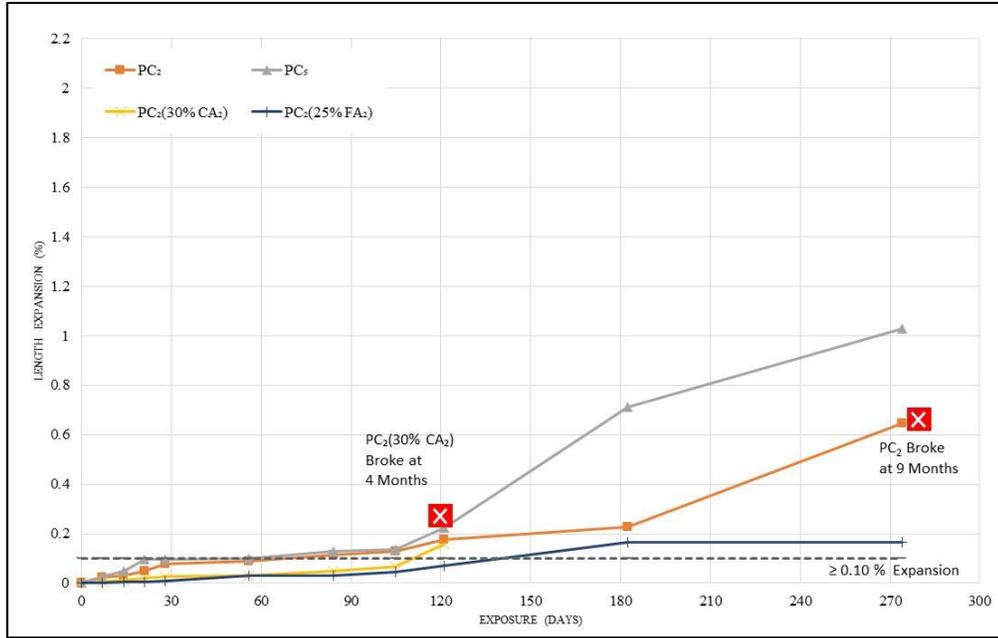


Figure 11. Expansion of the PC<sub>2</sub> & PC<sub>5</sub> based cement for the vacuum impregnation test

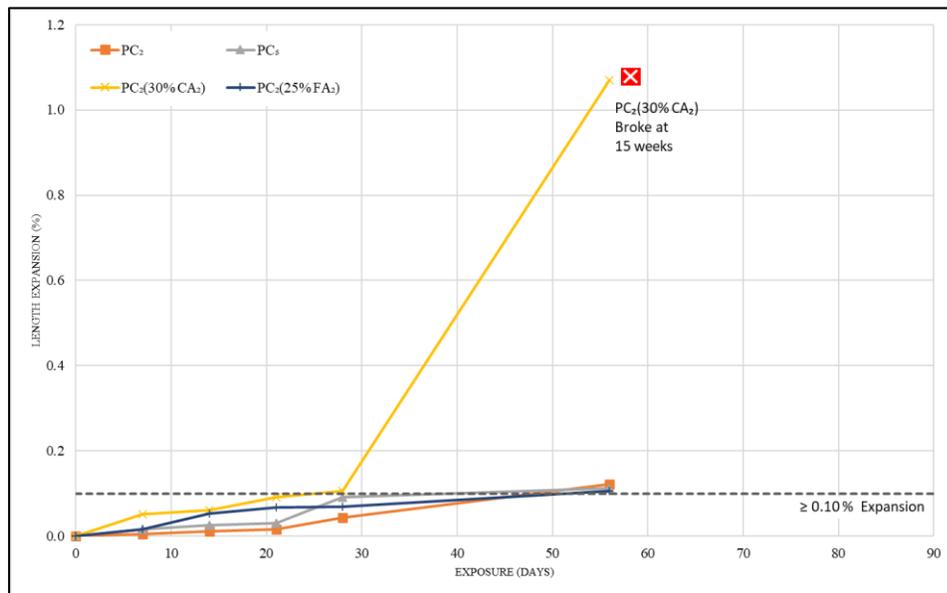
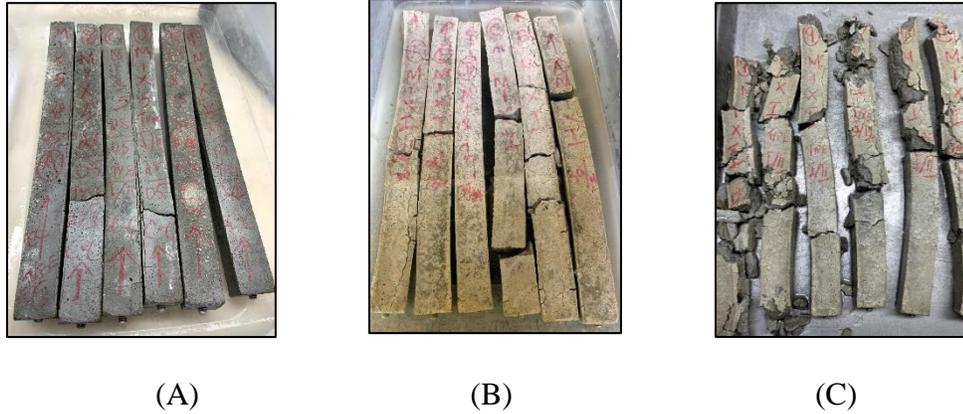


Figure 12. Expansion of the PC<sub>2</sub> & PC<sub>5</sub> based cement for the pH-controlled test

The measured length change according to ASTM C1012 is believed to assume that the whole specimen is expanding at the same time. However, Ferraris et al. observed that only a small fraction around the pin and ends of the specimen is responsible for the

bulk of the measured expansion. (Ferraris, Stutzman, Peltz, and Winpighler, 2005). Also, the results from the study done by Aguayo suggest that the vacuum saturation procedure is penetrating enough sulfate ions beyond the surface and throughout the center of the mortar specimen to cause early expansion. In addition, the accelerated penetration due to the vacuum impregnation technique and diffusion of sulfate ions is likely increasing the percentage affected by sulfate attack throughout the specimen, improving the “real” measured linear expansion of the mortar prisms. (Aguayo, 2019) Additionally, when the mortar prisms are placed in 5% Na<sub>2</sub>SO<sub>4</sub> solution where the pH of the solution is controlled around 7 (+/- 0.5) which is considered the ideal field condition the sulfate attack was aggressive. A pH-controlled environment sulfate test assures a test condition which is invariant with time and thus eliminates an important source of variability. As per the study done by Brown he developed a test and investigated the effects of pH of the sulfate solution on strength and expansion. He found that a control of the pH at 7 significantly increased the rate of sulfate attack, as measured by either strength loss or expansion, compared to the standard ASTM C 1012. (Brown, 1981). Thus, evidently the expansion rate of the mortar bars is highly accelerated indicating that the sulfate attack is quickened thereby drastically reducing the time at which the results are achieved. Clearly making it the most accelerated method among the other two methods. Specimens tested as per standard ASTM C1012, typically observed scaling and minor deterioration at the surface and along the edges before any significant expansion was recorded. Thereafter, it appears that cracking of the mortar bars has initiated, and the mechanism is controlled by the diffusion-reaction phenomenon (Gollop and Taylor, 1992) Figure 13 shows the

difference in level of severity and damage from the use of the pH-controlled procedure to accelerate the breaking from sulfate attack.



(A) (B) (C)  
Figure 13. Visual deterioration of control mixture (PC<sub>2</sub>) tested according to (A) ASTM C1012 after 1 year (B) Vacuum impregnated method around 9 months and (C) most accelerated pH-controlled method around 4 months in 5 % Na<sub>2</sub>SO<sub>4</sub> solution.

PC<sub>1</sub> seemed to have the most control in terms of constant forms of deterioration across all test methods. There did not appear to be a dramatic change in the measurements, deterioration, or compression strength as compared to other mixes.

### 6.3 Discussion on the time to failure and mass gain/loss of the mortar bars

Clearly the figure 14 indicates that for all control mixtures PC<sub>1</sub>, PC<sub>2</sub> & PC<sub>5</sub> (no SCM), the time to failure for mortar bars tested using the pH-controlled method was around 3 weeks, 8 weeks, and 10 weeks, whereas those tested according to the vacuum method was 8 weeks, 12 weeks, and 4 months and the controlled method showed a prolonged period of 8 weeks, 7 months and 9 months in solution for cements PC<sub>1</sub>, PC<sub>2</sub>, and PC<sub>5</sub>, respectively. As expected, a good correlation to resist sulfate attack was seen in the performance for each control mixture. Thereby clearly showing that based on their

C<sub>3</sub>A content for low, moderate, and high-sulfate resistant cements for the PC<sub>1</sub>, PC<sub>2</sub>, and PC<sub>5</sub> cement, respectively is evident. When the vacuum test provided results 2-3 times faster than ASTM C1012 the most accelerated pH-controlled method showed even more aggression as expected and yielded results within 8 to 10 weeks across all the three cements. Evidently PC<sub>2</sub> (25% FA<sub>2</sub>) i.e., the type II cement with 25% class F fly ash has performed well and indicates that the addition of F ash aids longer and higher sulfate resistance.

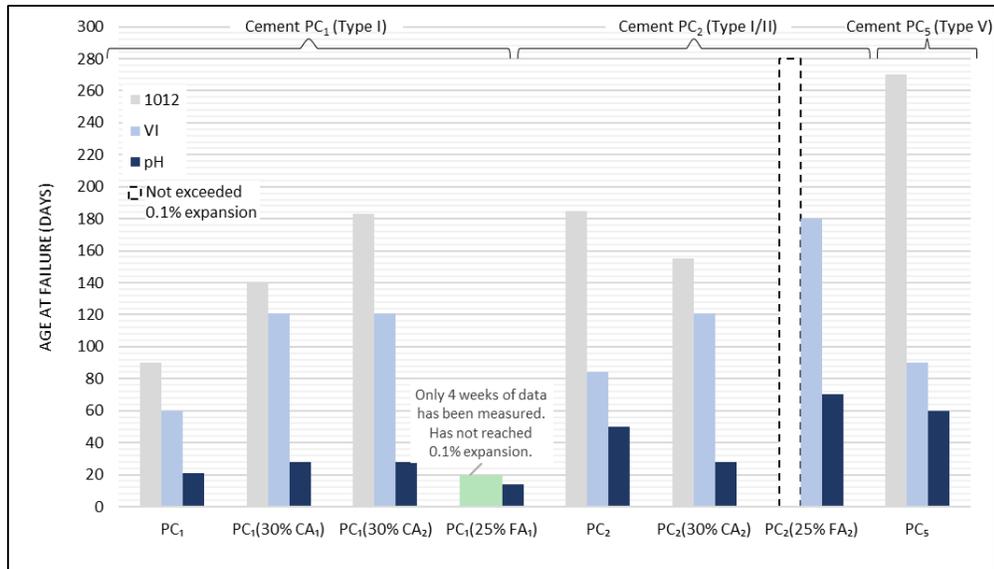


Figure 14. Time to failure ( $\geq 0.10\%$  expansion or fracture) submerged in 5% Na<sub>2</sub>SO<sub>4</sub> comparing across all the three tests methods.

The figure 15. represents the mass gain/loss of mortar bars pre- and post-vacuum impregnation for the pH-controlled test (most accelerated method). The measured mass loss in the mortar bars is associated with the drying regime in which specimens are placed in an oven at 38°C for 14 days prior to vacuum saturation to allow space and the ingress of Na<sub>2</sub>SO<sub>4</sub> solution in the now empty pores. The measured mass pre-vacuum and

post-vacuum saturation were nearly even for all mixtures with an average mass loss and gain of 6.2% and 6.5% respectively, between all mixtures.

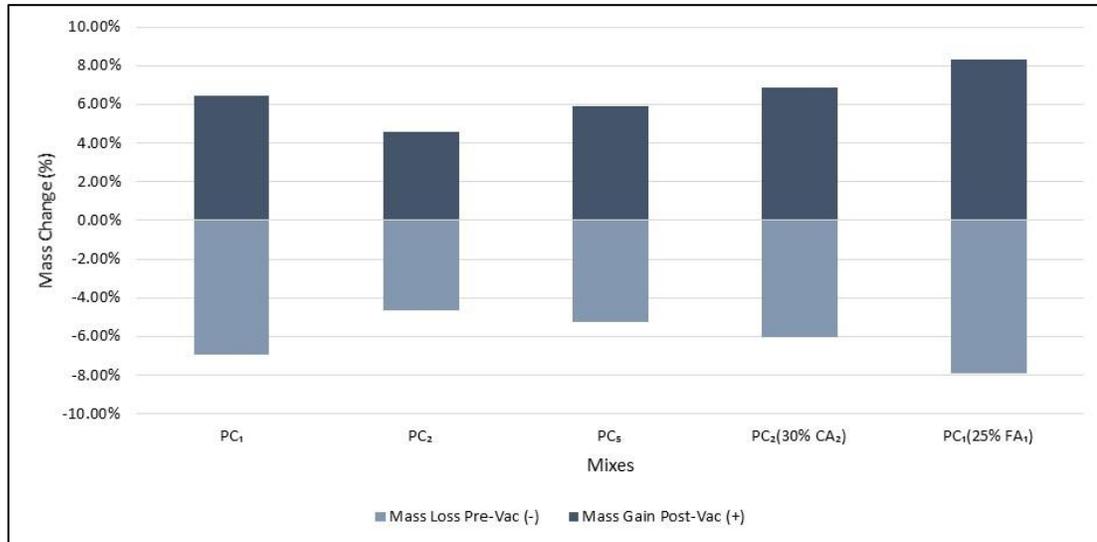


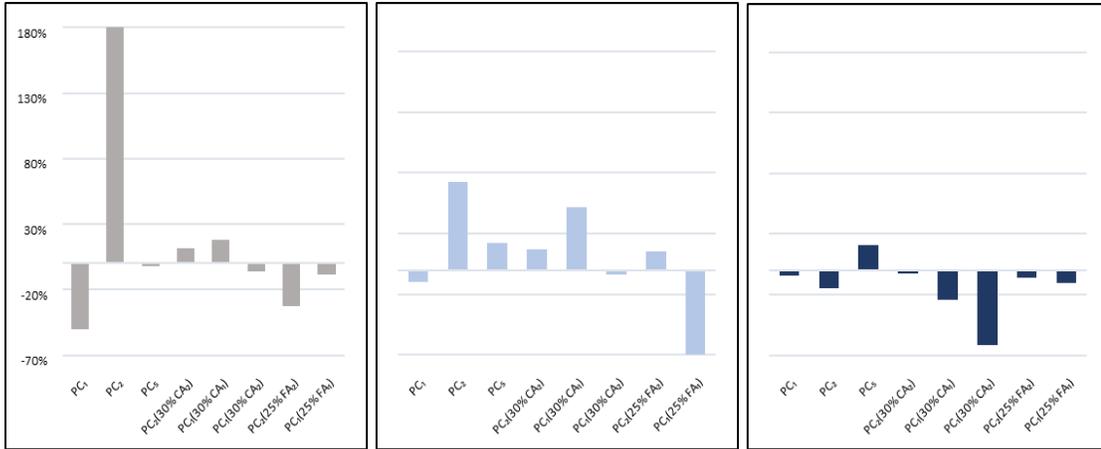
Figure 15. Mass gain/loss of mortar bars pre- and post-vacuum impregnation for the pH-controlled test (most accelerated method).

#### 6.4 Discussion on the compressive strength change across all the three mixes

The change in strength is calculated as a percentage of the strength of each cement paste measured after 7 days of curing. Loss in strength during the exposure period can be the result of cracking caused by expansion of ettringite and gypsum formation and the loss of C-S-H. Some cements may continue to hydrate in the sulfate solution and may experience an increase in strength. Susceptibility to sulfate attack can only be interpreted through strength loss. Increases in strength do not provide any information about sulfate resistance; such results only show that the cement continues to hydrate in sulfate solution over the test period. (Paulo, Jeffery, Kimberly, and John, 2000) In contradictory to the study there is an observation that there is some increase in the strength from ettringite

crystal growth. At least in my opinion, as ettringite crystals grow they also fill pores in the concrete which are typically empty initially especially early on (8 weeks) and if filled with solid material (i.e., ettringite) would actually serve as an increase in strength. However, I do agree that ultimately given time, cracking, and further damage you should see a strength decrease and this is ultimately what we are capturing as sulfate performance.

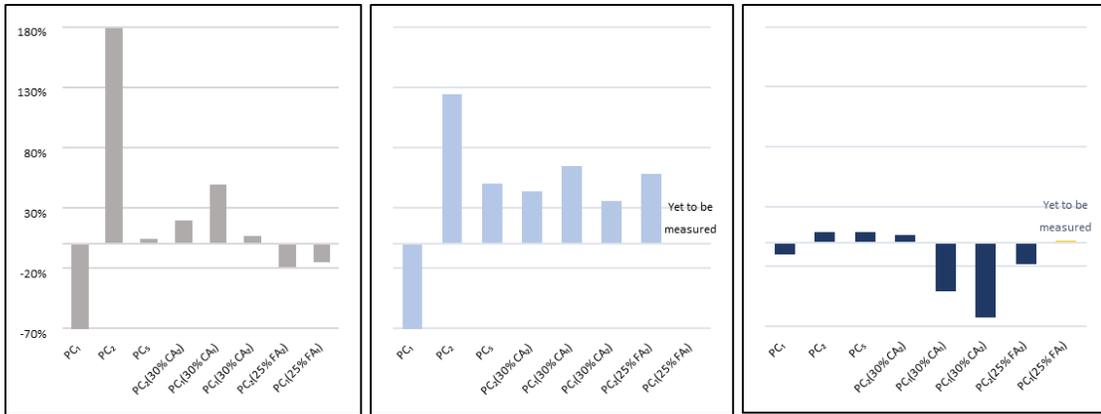
Figure 16 shows the effect of 8 weeks (56 days) of exposure to 5% sodium sulfate solution on the compressive strength, as compared to initial strength measured after 7 days of curing across all the three test methods. The PC<sub>1</sub> (Type I) cubes have consistently lost their strength across all the three tests but the PC<sub>2</sub> (Type I/II) has lost strength only in the most accelerated pH-controlled method. At this 8<sup>th</sup> week measurement, the PC<sub>5</sub> (Type V) cubes have gained strength around 25% to 27% from the initial 7-day strength. Looking into the performance of class C and class F fly ashes, it's contradictory to observe that the class C fly ash has seen a consistent strength gain when it comes to the standard C1012 method and vacuum method, whereas the class F fly ash seem to have considerably lost strength up to 70% in the vacuum method. But in the pH-controlled method it seems that all the cements lost strength except PC<sub>5</sub> (Type V) cement which is considered to have high sulfate resistance.



(A) Controlled (ASTM 1012)      (B) Vacuum Impregnation      (C) pH-Controlled

Figure 16. Results of comparing the compressive strength change after 8 weeks (56 days) of exposure. Change in strength is based upon initial strength measured at 7 days of curing, prior to exposure, across all the three tests.

Figure 17 shows the effect of 12 weeks (84 days) of exposure to 5% sodium sulfate solution on the compressive strength, as compared to initial strength measured after 7 days of curing across all the three test methods. Similar to the 8<sup>th</sup> week exposure PC<sub>1</sub> (Type I) cubes have lost their strength across all the three tests but the PC<sub>2</sub> (Type I/II) has lost strength only in the most accelerated pH-controlled method. And the PC<sub>5</sub> (Type V) cubes have gained strength on an average 25% to 30% from the initial 7-day strength. The class C ashes have performed well and show strength gain in both the controlled and the vacuum method, but in the pH-controlled method the cubes have considerably lost strength. Whereas when we consider the class F fly ash we have one mix PC<sub>2</sub> (25% FA<sub>2</sub>) (type I/II cement with 25% of F ash) which does not show a constant pattern of loss or gain instead it seems to be varying across all the three tests.

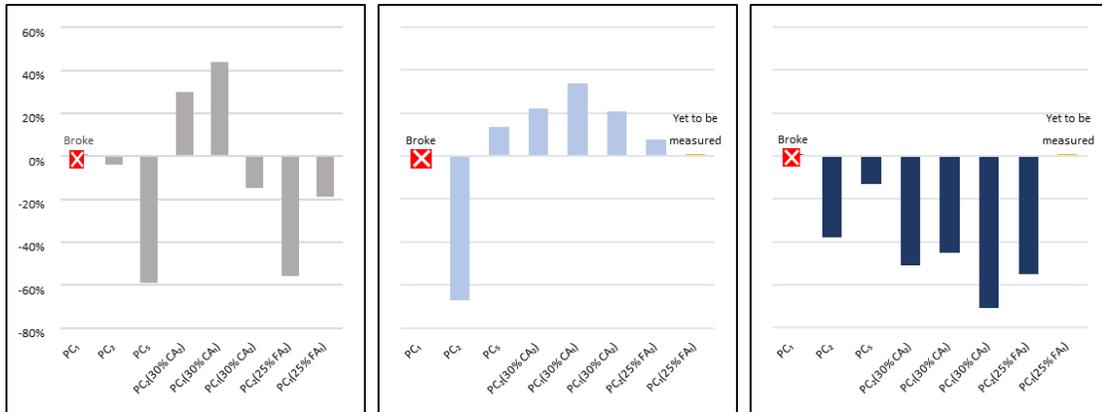


(A) Controlled (ASTM 1012)      (B) Vacuum Impregnation      (C) pH-Controlled

Figure 17. Results of comparing the compressive strength change after 12 weeks (84 days) of exposure. Change in strength is based upon initial strength measured at 7 days of curing, prior to exposure, across all the three tests.

Figure 18 shows the effect of 15 weeks (105 days) of exposure to 5% sodium sulfate solution on the compressive strength, as compared to initial strength measured after 7 days of curing across all the three test methods. And clearly the PC<sub>1</sub> (Type I cement) which is the least sulfate resistant was completely broken across all the three tests, thereby it could not be measured. Since cubes have been exposed to the 5% Na<sub>2</sub>SO<sub>4</sub> under a pH-controlled environment where the pH and sulfate concentration of the solution are maintained throughout the test period, all the cubes have uniformly lost their strength thereby clearly proving that the pH-controlled method is the most accelerated method and depicts highest sulfate attack in comparison with the other two methods. Looking into figure 17 (A) (B) & figure 18 (A) (B) it seems that there is not a very big difference between the 12<sup>th</sup> week and 15<sup>th</sup> week cube strength. Ideally the PC<sub>2</sub> (Type I/II)

has clearly lost its strength upto 60% to 70% while comparing the 12<sup>th</sup> week and 15<sup>th</sup> week data.



(A) Controlled (ASTM 1012)      (B) Vacuum Impregnation      (C) pH-Controlled

Figure 18. Results of comparing the compressive strength change after 15 weeks (105 days) of exposure. Change in strength is based upon initial strength measured at 7 days of curing, prior to exposure, across all the three tests.

Some of the researchers have recommended using a 25% to 30% reduction in original strength as an indication of poor sulfate resistance (Mehta, 1986), (Cohen, 1988).

### 6.5 Summary of the compressive strength of mortar bars

From the 8<sup>th</sup> week, 12<sup>th</sup> week and 15<sup>th</sup> week sulfate resistance test results, performance of the 8 mixes with variations specifically, for the pH-controlled method can be described by the following ranking:

At 8<sup>th</sup> week (56 days),

PC<sub>5</sub> (Type V) > PC<sub>2</sub> (30% CA<sub>2</sub>) > PC<sub>1</sub> > PC<sub>2</sub> (25% FA<sub>2</sub>) > PC<sub>1</sub> (25% FA<sub>1</sub>) PC<sub>2</sub> > PC<sub>1</sub>  
(30% CA<sub>1</sub>) > PC<sub>1</sub> (30% CA<sub>2</sub>)

At 12<sup>th</sup> week (84 days),

PC<sub>5</sub> (Type V) > PC<sub>2</sub> > PC<sub>2</sub> (30% CA<sub>2</sub>) > PC<sub>1</sub> > PC<sub>2</sub> (25% FA<sub>2</sub>) > PC<sub>1</sub> (25% FA<sub>1</sub>) PC<sub>1</sub>  
(30% CA<sub>1</sub>) > PC<sub>1</sub> (30% CA<sub>2</sub>)

The 15<sup>th</sup> week strength loss need not be mentioned exclusively, since all the mixes have completely lost strength also all the mixes show reduction in strength above 25%

Overall, the test results show the sulfate resistance of these cements should be examined individually because no general trends were apparent based upon cement classification. Monosulfate hydrate and calcium hydroxide are the cement hydration products that react directly with sulfate ions. The volumes of these products formed during cement hydration are directly linked to the chemical composition of the cement. (Paulo, Jeffery, Kimberly, and John, 2000). The chemical composition may vary substantially within each class of cements. The sulfate resistance of Type V cement is considered the baseline evaluation of all the other cements in this research.

## 7. CONCLUSIONS AND RECOMMENDATIONS

### 7.1 Conclusion

This research presented a new proposed test method which aimed at significantly reducing the duration of the ASTM C1012 by accelerating the penetration and diffusion of sulfates in mortar specimens placed under high vacuum and by exposing it to a pH-controlled environment. Also the evaluation of the sulfate performance of mortar bars and mortar cubes by measuring the expansion or the length change in the mortar bars and measuring the compressive strength change of the mortar cubes across three test methods. Additionally, the research presents evaluation and access performance on a range of mixtures varying in chemistry including different fly ashes and different types of cement.

The pH-controlled method consistently observed higher expansion and aggressive strength loss when compared with vacuum impregnation and ASTM C1012 at 5 % Na<sub>2</sub>SO<sub>4</sub>. Since the mortar bars and cubes undergo the vacuum impregnation method in which the sulfate ions are accelerated and diffused into the specimens thereby not just affecting the external surface causing cracks but improves the linear expansion of the bars and the strength of the cubes. Additionally, the pH of the solution was maintained constant similar to the real field scenario, which would aggravate the sulfate attack and provide results 2-3 times faster than the vacuum impregnation method.

The visual deterioration between comparing the mortar bars and mortar cubes across all the three tests clearly indicate that larger cracks and a quick complete disintegration of the specimens occurred rapidly for the mortar bars and cubes subjected to the pH-controlled method, thereby making it the most accelerated method among the

other two tests. The inference from the compressive strength test results show that the sulfate resistance of these cements should be examined individually because the tests did not show any general trends based upon cement classification.

## **7.2 Recommendation for Future Work**

Repeatability between the observed expansion as well as some of the recent redo mixtures for the test method are still ongoing and require further consideration. Ideally, even more variations in the fly ashes can be tested, also to yield more field related results, concrete mixes need to be tested using the same three test methods. Lastly, different sulfate solutions like the  $\text{MgSO}_4$  can be used along with the  $\text{Na}_2\text{SO}_4$  to compare the impact of the sulfate solution.

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