

# A Comprehensive Review on the Mechanism of Concrete Deterioration in Accelerated Aggressive Environment

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doi: <https://doi.org/10.21467/proceedings.112.40>

## ABSTRACT

Concrete is being extensively utilized for the constructional and other allied works on account of its versatility and mechanical properties. However, it exists to be in a state of disequilibrium with its ambient environment owing to its universal alkaline nature and hence is susceptible to deterioration when exposed to aggressive environments. The reactive species emanating from chemical plants, fertiliser industries, marine water, agro-food industries etc., produce detrimental effects on the concrete structures through the dissolution of calcium bearing phases from the hydrated matrix. This degradation culminates in decalcification, volumetric expansion, salt crystallisation, micro-cracking, surface scaling, delamination, spalling and corrosion. Diffusivity, capillary porosity, permeability, chemical nature of hydrated matrix and pore network are the parameters that influence the chemical mechanism of concrete degradation. The mechanism of concrete degradation is distinct for various aggressive species and its fair comprehension remains as one of the challenges in accomplishing the durability based concrete design. This paper critically reviews the basic mechanism of the concrete deterioration in accelerated aggressive environment of mineral acids, organic acids and inorganic salts. In addition to this, a glimpse of the effect of degradation on different binder systems viz., Ordinary Portland Cement system, blended cement system, special cement system and alkali activated system is provided.

**Keywords:** Concrete, deterioration, aggressive species, mechanism, decalcification, inorganic acid attack, organic acid attack, inorganic salt attack and binder systems

## 1 Introduction

The rise in demand of concrete on account of its mechanical characteristics and flexible nature is vivid from the remarkable per annum global production that exceeds 10 to 25 billion tonnes. Nevertheless, its predominant alkaline nature has made it liable to the aggressive species attack, emanating from various sources of salts and acids. Consequently, premature deterioration of concrete structures is visible within few years of commissioning and hence its design service life is reduced. In India, the concrete structures designed for a service life of 70-80 years are functional only for a period of 30-50 years. Fifty percent of the structures are subjected to repairs in about 10 year span. Around 50% of the construction budget is spent on maintenance and retrofitting of structures in degraded state. The statistical reports of 2016 revealed that India losses around 4-5% of Gross Domestic Product (GDP) annually on corrosion related issues. Hence concrete deterioration is a grave issue to be contemplated.

Concrete deterioration can occur both physically and chemically. The deterioration of concrete by physical mechanism includes shrinkage, thermal cracking, freeze-thaw attack, abrasion and erosion. The chemical mechanism include the acid attack (both organic and inorganic acids), carbonation, alkali-aggregate reaction



(AAR) and attack by different salts (sulphates, chlorides and nitrates) (Dyer, 2014). The concrete deterioration by chemical reactions can be classified into three viz., exchange reactions between aggressive fluid and components of hydrated cement paste (HCP), reactions involving hydrolysis and leaching of components of HCP and reactions involving the formation of expansive products (Owens, 2009). Generally, in Portland cement systems the concrete degradation starts when the aqueous solution of aggressive species replaces the sodium and calcium ions in the pore water of the hydrated matter. Portlandite decomposes initially as the pH of the pore water marginally decrease. Further reduction in pH causes the calcium monosulphoaluminate ( $AF_m$ ) and ettringite/Calcium trisulphoaluminate hydrate ( $AF_t$ ) to dissolve and compounds such as amorphous silica, amorphous alumina and iron hydroxides get precipitated. Thus the Ca/Si ratio considerably reduces and decalcification of the C-S-H gel (calcium silicate hydrate) occurs. Hence the binding property and strength is adversely affected (Dyer, 2017a).

Since the comprehension of degradation mechanism is the key for formulating prevention and mitigation measures, it could be accomplished by the micro-analytical studies viz., X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Mercury Intrusion Porosimetry (MIP), Thermo-Gravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), Scanning Electron Microscopy-Electron Dispersive Spectrometry (SEM-EDS), Nuclear Magnetic Resonance (NMR) etc (Scrivener et al., 2016). This would provide sufficient information on the chemical composition, morphology, impurity composition, associated physical events, qualitative and quantitative analysis of products formed due to chemical interaction. As degradation mechanisms are influenced by the transportation characteristics of cementitious matrix, empirical models could be formulated by incorporating transport parameters that could shed light on degradation kinetics and also in service life prediction. This paper critically reviews chemical mechanism of concrete degradation subjected to acid and salt attack, and also outlines the effect of aggressive species on different binder systems.

## **2 Chemical Mechanism of Concrete Deterioration**

Concrete deterioration by chemical means is influenced by the service environment of the structures. The reactive species generated by mineral acids, organic acids and salts have different chemical mechanism of degradation and are as discussed further.

### **2.1 Acid Attack**

The acids that pose threat to concrete structures could be organic or mineral acids and are described below in detail. Three mechanisms are primarily involved in the process of concrete deterioration subjected to acid invasion viz., acidolysis, complexolysis and precipitation of the products formed. The basic mechanism of attack for all the acids is acidolysis. For organic acids, all the three mechanisms are found to occur simultaneously. Complexolysis is influenced by the existence of metal ions in the solution, which react with the acid ions to form complexes. Precipitation is not always detrimental, except when the molar volume of salts is greater than that of the hydration products (Dyer, 2017c).

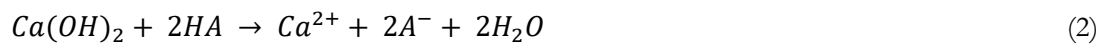
#### **2.1.1 Mineral Acids**

Sulphuric acid, hydrochloric acid, nitric acid, phosphoric acid and carbonic acid are intense enough to promote the degradation process. The sources of these acids could be natural water, gaseous air pollutants, acid rain and industrial effluents (Alexander et al., 2013). The chemical reactions shown below provides the mechanism of acid action on hardened concrete.

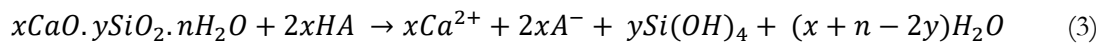
Dissociation of Acid:



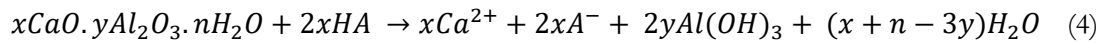
Acid attack on Calcium Hydroxide:



Decomposition of C-S-H gel (pH~10.7):



Decomposition of C-A-H:



Varied end results are exhibited by the invasion of different mineral acids on concrete. The nitric acid attack creates a soft corroded layer with cracks which consist of two zones. The white coloured (first) zone is dominated by the presence of SiO<sub>2</sub> whereas the brown coloured zone (second zone) contains ferric hydroxide besides silicon dioxide. The volume reduction of the corroded layer is bound to occur due to the leaching of calcium nitrate formed as a byproduct besides amorphous gel of Si, Al and Fe. The interaction of hydrochloric acid and concrete develops a three layered zone. The first zone composes of aggressive materials and cement paste components. The other two zones are predominated by the corrosion products (Alexander et al., 2013). The microorganisms such as thiobacillus oxidises the hydrogen sulphide in the sewers (Figure 1) to sulphuric acid which further acts on the concrete to form gypsum. Gypsum causes expansion of concrete and induces cracks. Decalcification of the cementitious matrix due to the sulphuric acid attack causes considerable strength reduction. Elemental sulphur (S<sup>0</sup>) and thiosulphates (S<sub>2</sub>O<sub>3</sub><sup>2-</sup>) are the intermediates formed during the reaction (Khan et al., 2019).

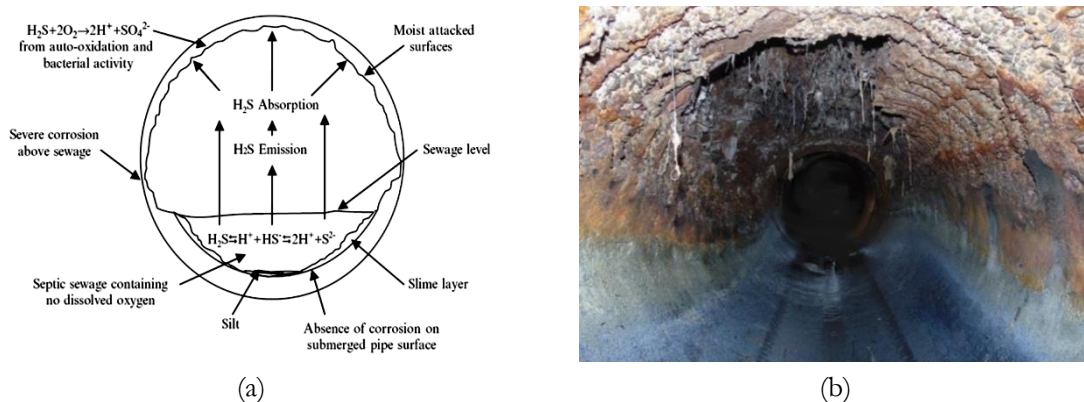


Figure 1. (a) Degradation of sewer pipelines on sulphuric acid exposure (Alexander et al., 2013)

(b) Microbially Induced Concrete Corrosion of sewer in Rockland County, New York

(<https://www.wwdmag.com/pumpscentrifugal/microbiologically-induced-corrosion>)

### 2.1.2 Organic Acids

Citric acid, acetic acid, succinic acid, butyric acid, oxalic acid, tartaric acid and malic acid are the common organic acids. These acids emanate from the agricultural effluents such as liquid manure and silage effluent, agrofood effluents such as dairy effluents, whey and white water, sugar beet byproducts, sugar cane byproducts and effluents from fermenting industries such as winery waste water, sugar distillers and water from malt industry. The mechanism of organic acid attack is same as that of mineral acids, except the dissociation process. Partial dissociation of ions is observed in organic acids while complete dissociation is exhibited by mineral

acids. Albeit these acids are weak in nature, it triggers the degradation action owing to its poly acidic behaviour (Ramaswamy and Santhanam, 2018). The aggressiveness of organic acids are governed by dissociation constant of acid, poly acidity, pH of solution, solubility of salt formed, molar volume of salt and complexation of cations, mesoscopic shape of salt and kinetics of salt formation (Larreur-Cayol et al., 2011) , (Alexander et al., 2013). The aggressiveness of various acids in the increasing order is Oxalic < Tartaric < Malic < Acetic < Succinic < Citric acid. The action of some organic acids on the concrete is discussed below.

Acetic acid ( $CH_3COOH$ ) is moderately aggressive with a dissociation constant ( $pK_a$ ) of 4.76 (at 25 °C) and hence is monobasic. Highly soluble calcium acetate monohydrate ( $Ca(CH_3COO)_2 \cdot H_2O$ ) is the salt precipitated from the reaction. The other salts namely aluminium acetate and ferric acetate is less soluble, which reduces the shrinkage of the corroded layer formed and leads to lower rate of corrosion compared to citric or succinic acid attack (Dyer, 2014). The attack results in almost complete decalcification of the matrix and dissolution of hydrated and anhydrous phases of the cement matrix (Ukrainczyk et al., 2019). The deterioration reactions due to the acetic acid interaction is as follows (Larreur-Cayol et al., 2011):



Citric Acid ( $C_6H_8O_7$ ) is a very highly aggressive acid with an acid dissociation constant of 3.14/4.77/6.39 (at 25 °C). Slightly soluble calcium citrate tetrahydrate ( $Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$ ) is the byproduct formed. It possesses higher molar volume that prompts the breaking of the outer portion of the cementitious matrix. The dissociation reactions involved in citric acid is as given in equation 8, 9 and 10 (Larreur-Cayol et al., 2011).



Succinic acid ( $C_4H_6O_4$ ) is a highly aggressive acid with a dissociation constant of 4.16/5.61(at 25 °C) which produces calcium succinate trihydrate, a slightly soluble salt on reaction with hydrated matrix. The transition zone (zone between inner core and degraded zone) is supplemented with  $SO_3$  that hastens ettringite formation. Inner part of degraded zone is enriched with the precipitated salt whereas the outer portion contains the amorphous Al and Fe bearing silica gel (Ninan et al., 2020).

Oxalic acid ( $C_2H_2O_4$ ) is less aggressive with a dissociation constant of 1.23/4.19 (at 25 °C). The insoluble calcium oxalate monohydrate is formed on the surface without calcium ion loss, as a result of its attack. The salt protects the cement matrix by hindering the ingress of aggressive species into the matrix.

Tartaric acid ( $C_4H_6O_6$ ) with an acid dissociation constant of 3.04/4.37 (at 25 °C) has a moderately low aggressive nature. Calcium tartarate tetrahydrate ( $CaC_4H_4O_6 \cdot 4H_2O$ ) is the salt precipitated on the surface of the concrete. It protects the matrix for a limited duration due to its slightly soluble nature. An unaltered inner core and a transition zone is present in the matrix, similar to the other acids. Calcium malate precipitates in the outer region of the specimen when subjected to malic acid ( $C_4H_6O_5$ ) attack. The mechanism of attack is similar to that of the tartaric acid attack. Apart from these, the other organic acids with deterioration capacity include lactic, butyric, glycolic, catechol, propionic, aconitic and fumaric acid.

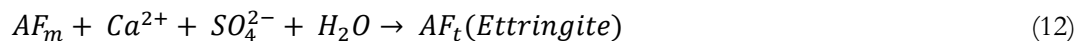
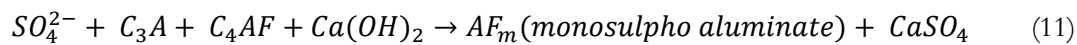
## 2.2 Inorganic Salt Attack

Inorganic salts of sulphates, nitrates, chlorides and phosphates critically influence the mechanism of concrete degradation. The mechanism of attack of sulphates, chlorides and nitrates are elucidated in detail.

### 2.2.1 Sulphate Attack

Sulphate attack could be internal due to delayed ettringite ( $AF_t$ ) formation or external due to increased sulphate concentration in the environment. Sulphate attack culminates in the formation of gypsum ( $CaSO_4$ ) and ettringite or modification of the C-S-H structure.

External sulphate attack is the most explored deterioration process in concrete, caused when sulphates reacts with the hardened concrete which eventually gives rise to cracks, excessive expansion, surface spalling, delamination and deprivation of mechanical properties. The sulphate ions from ground water, surface water, sea water, waste water treatment plants and natural soils react with the hydrated aluminates to form ettringite which is poorly water soluble and causes increase in the volume. The primary ettringite formed as a consequence of sulphate ions from the gypsum while the concrete is in its plastic state, is not significantly expansive. However, the reactive alumina from the monosulphate ( $AF_m$ ) or unreacted  $C_3A/C_4AF$  present in the hydrated matrix of hardened concrete reacts with the external sulphates to form  $AF_t$ , that can generate considerable expansion. Equation 11 and 12 shows the products formed as a result of the sulphate attack (Alexander et al., 2013).



The deterioration mechanism of sulphates salts varies due to the presence of different types of associated cations, which is provided in Table 1. The microstructure of the products formed by sulphate attack is shown in Figure 2.

Table 1. Deterioration mechanism of different sulphate salts (Alexander et al., 2013)

Salt	Solubility @ 20°C (g/100ml)	Reactant	Product	Associated Phenomena
$CaSO_4$	0.255	$AF_m, C_3A, C_4AF$	$AF_t$	Cracks due to expansion
$Na_2SO_4$	19.5	$Ca(OH)_2$ , Alkali	$AF_t$ , Secondary gypsum, U phase	Chance of alkali aggregate reaction, Microcracking, shrinkage and surface scaling
$K_2SO_4$	11.1			
$MgSO_4$	62.9	$Ca(OH)_2$ , CAH, CSH	Brucite, Secondary gypsum, $AF_t$ , Magnesium silicate hydrate, aq. silica	Expansion and disintegration
$(NH_4)_2SO_4$	75.4	$Ca(OH)_2$ , CSH	Gypsum, $NH_3$ , aqueous silica	Expansion and softening of matrix

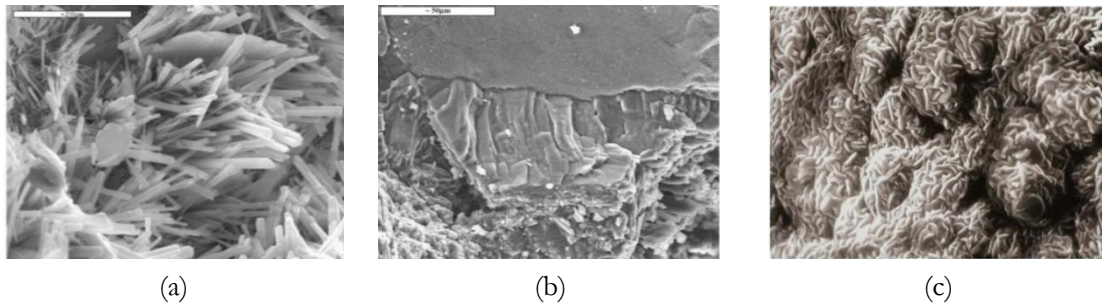


Figure 2. Microstructure of (a) Ettringite (b) Gypsum crystallised in aggregate-paste interface (c) Brucite formation due to magnesium sulphate (Alexander et al., 2013)

### 2.2.2 Nitrate Attack

Ammonium nitrate is the major nitrate salt that cause deleterious effects on the cement based materials. It is abundantly utilized in the fertilizer plants and domestic feed. Ammonium nitrate attack terminates in the formation of highly soluble calcium nitrate, slightly soluble calcium nitro-aluminate and aqueous ammonia. The end result is the leaching of calcium hydroxide followed by the gradual decalcification of C-S-H gel. Dissolution of the compounds from the matrix accompanied by diffusion or crystallization are the steps involved in the mechanism of ammonium nitrate invasion (Schneider, 2009; Alexander et al., 2013; Arafa et al., 2015).

### 2.2.3 Chloride Attack

Sodium chloride, magnesium chloride and ammonium chloride are the main chloride salts that accelerate the concrete degradation. Chloride anions react with the tricalcium aluminate in the matrix to form chloroaluminates such as Friedal's salt ( $\text{Ca}_4\text{Al}_2(\text{OH})_{12}\text{Cl}_2 \cdot 4\text{H}_2\text{O}$ ), which is non expansive in nature. When chlorides are present in high concentration, the expansive behaviour of ettringite produced by sulphate attack is diminished. Albeit chlorides reduce ill-effects of ettringite, its ingress can be pernicious to the steel reinforcement. If chloride content at the reinforcement surface exceeds a specific threshold value, the passivating layer surrounding the steel gets destroyed and corrosion of reinforcement occurs. Subsequently, the formation of corrosion products occur and the load bearing capacity of the reinforced concrete is threatened (Ragab et al., 2016). Apart from Friedal's salt, Kuzle's salt ( $\text{Ca}_4\text{Al}_2(\text{OH})_{12}\text{Cl}(\text{SO}_4)_{0.5} \cdot 5\text{H}_2\text{O}$ ) which hinders the ettringite expansion is also produced. Due to the combined interaction of chlorides and sulphate with the cementitious matrix, the whole process becomes intricate. This is called interaction or synergy effect (Cao et al., 2020).

## 3 Effect of Aggressive Species on Different Binder Systems

The action of the aggressive species on various binder systems is different. It is mainly governed by the binder composition and the products formed during the hardening of the concrete. The binder systems included in this section are OPC system, OPC blended system and alkali activated systems.

Miletić et al. (1999) assessed the effect of sulphates by exposing the Portland cement (PCB) and Portland flyash cement (PCBP) samples to 10% ammonium sulphate solution for 3, 7, 14, 21 and 28 days. Substantial quantity of gypsum was observed in the samples attacked by ammonium sulphate whereas ettringite formed was of lesser quantity. XRD test results (Figure 3) revealed that gypsum content was more in exposed PCBP samples than PCB samples. The sulphate resistance of PCBP cement could be attributed to the lesser amount of tricalcium aluminate and Portlandite, essential for the gypsum formation.

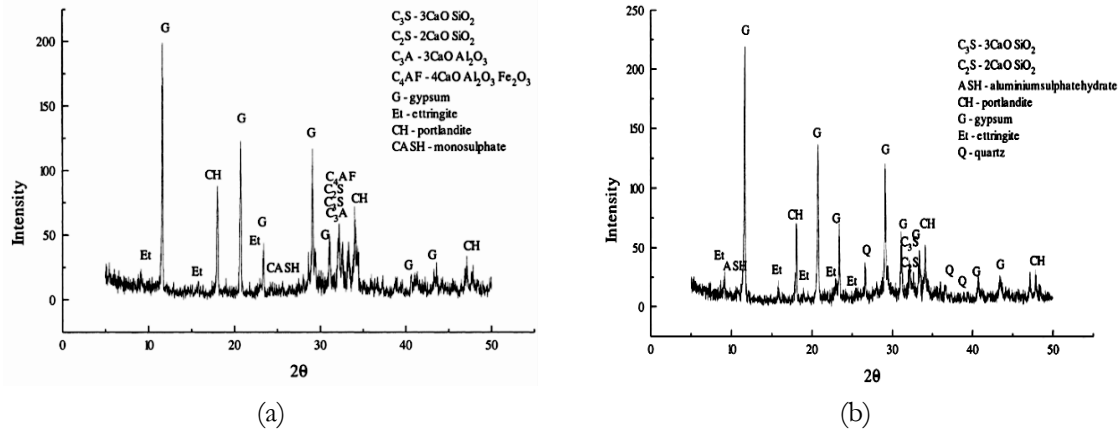


Figure 3. XRD pattern of paste samples after 21 days of exposure (a) PCB (b) PCBP

Fernandez-Jimenez et al. (2007) studied the resistance of alkali activated flyash (AAFA) under aggressive conditions of seawater, sodium sulphate, hydrochloric acid and deionized water. No significant deterioration of alkali activated flyash samples took place. The strength property increased irrespective of the medium of exposure (Figure 4). AAFA mortar with a mixture of NaOH and sodium silicate alkali exhibited greater resistance in different aggressive medium.

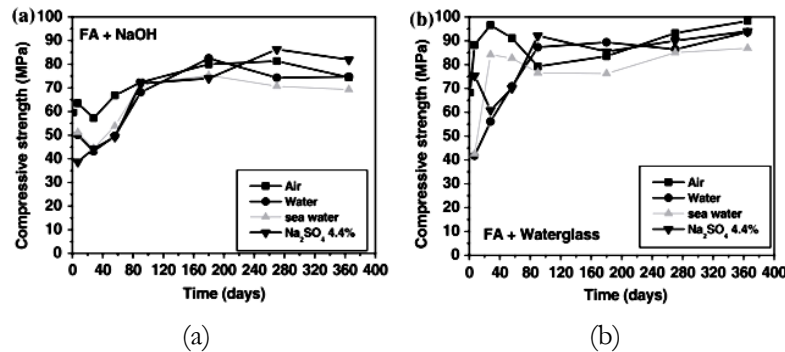


Figure 4. Compressive strength of (a) Flyash activated mortar in 8M NaOH (b) Flyash activated mortar in 15% Na<sub>2</sub>SiO<sub>3</sub> and 85% 12.5M NaOH (Fernandez-Jimenez et al., 2007)

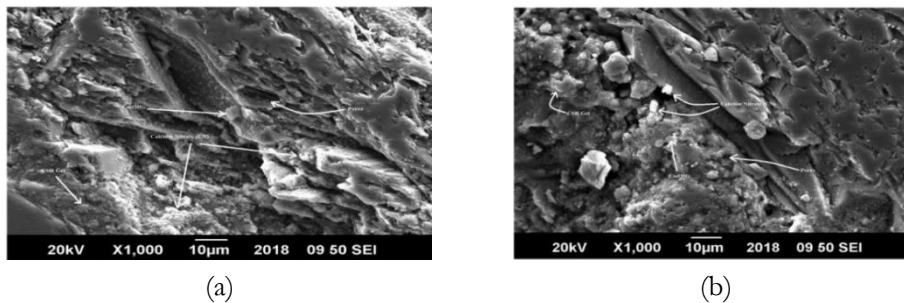


Figure 5. SEM images of samples (a) Control (b) SF composite concrete (Bibi et al., 2020)

Bibi et al. (2020) investigated the effect of 5% ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>) on the microstructure and strength of silica fume (SF) blended concrete. The concrete was partially replaced with SF for percentages 0, 5, 8 and 10. XRD and SEM tests results indicated that the addition of SF in concrete aids in reducing the calcium hydroxide and positively contributed towards the durability. The concrete specimens with SF content of 8% exhibited considerable enhancement in strength and showed higher resistance to NH<sub>4</sub>NO<sub>3</sub> attack compared to

the other concrete samples. Figure 5(a) shows the SEM image of control sample with visible calcium nitrate (CN) formation and large pores. Figure 5(b) is the SEM image of 8% SF composite concrete with least CN formation and less pores.

Dyer (2017b) evaluated the performance of different cement types (Portland cement (PC), blend of Portland cement and flyash (PC/FA) and calcium sulphoaluminate cement (CSA)) to acetic and butyric acid (0.1 M and 0.01M) attack. It was inferred that rate of mass loss is dependent on the solution concentration. At low acid concentration, the mass loss was higher for PC. At higher solution concentration, CSA showed highest mass loss. The increased mass loss of CSA paste is observed in acetic acid than the butyric acid.

Amin and Bassuoni (2017) conducted an experimental study on the resistance of the Portland limestone cement (PLC) with addition of the supplementary cementitious materials such as flyash, nanosilica and silica fume when exposed to 5% ammonium sulphate solution for 12 months. Microstructural analysis, mass loss, penetration depth and phenolphthalein tests revealed that binary binders consisting of 5% silica fume, 5% nanosilica and 30% flyash showed enhanced resistance towards ammonium sulphate attack. Hence it could be established that blended cement systems are capable of withstanding aggressive environment. Moreover, it is also an efficient method for the utilization of industrial byproducts so as to achieve sustainability.

#### 4 Conclusions

Concrete is a user-friendly material which is being extensively used for the construction practices since many decades. But it is prone to rapid deterioration and has negatively impacted the GDP of many countries. The chemical mechanism of concrete deterioration include acid attack, alkali aggregate reaction, carbonation and attack by different salts. The external aggressive species interact with the hydrated cementitious matrix and results in decalcification, volume expansion, cracking, delamination, spalling and corrosion. The mechanism of concrete degradation is caused by different aggressive species and varies with the service environment. All the mineral acids and inorganic salts cause intense deterioration. Carbonation and ingress of chloride ions ultimately culminates in the reinforcement corrosion, affecting the durability of structures. The weak organic acids cause deterioration only in specific environment and is influenced by their polyacidic character. From the researches undergone, it was found that citric acid is the most aggressive organic acid followed by succinic acid, acetic acid, malic acid, tartaric acid and oxalic acid. Studies indicate that OPC blended cementitious systems and alkali activated binder systems are more resistant to the aggressive species attack compared to conventional OPC system.

Since durability based design approach is gaining pertinence currently, it could be achieved only if the deterioration mechanism and alteration kinetics when exposed to various service environment are unravelled. A fair comprehension of microstructure and modelling of kinetics for the different binder systems exposed to various aggressive environments would promote the maturing of the performance based approach that could eventually lead to the construction of durable and strong structures with extended service life.

#### How to Cite this Article:

Sneha, S. S., & Ramaswamy, K. P. (2021). A Comprehensive Review on the Mechanism of Concrete Deterioration in Accelerated Aggressive Environment. *AIJR Proceedings*, 332-340.

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