

# Deterioration of alkali-activated mortars exposed to natural aggressive sewer environment

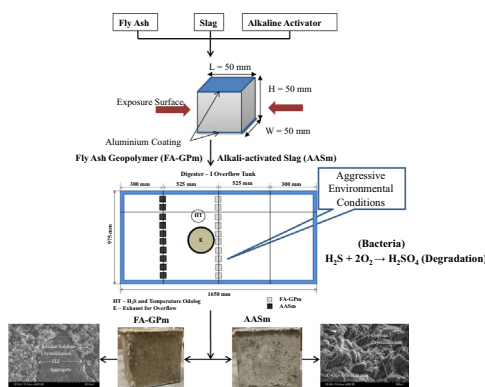
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## HIGHLIGHTS

- Alkali activated mortars were exposure to natural sewer environment.
- Visual and microstructural assessment was performed.
- Loss in surface pH and pH within the depth was observed.
- Thenardite and gypsum crystallization within the microstructure was observed.
- Sulphur concentration of both alkali-activated mortars was evaluated.

## GRAPHICAL ABSTRACT



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## ABSTRACT

This study aims to evaluate the performance of low calcium fly ash based geopolymer (FA-GPm) and alkali-activated slag based mortars (AASm) in an aggressive sewer environment. Specimens were extracted from field exposure, after 6 and 12 months. Visual, chemical and physical analyses were performed to evaluate the deterioration. Surface pH was evaluated to identify the microbial induced corrosion (MIC) stage in each specimen. The neutralization depth of the specimens was measured by using the phenolphthalein indicator. Extensive microstructural analyses were carried out to assess the extent and type of deterioration of gel matrix by using techniques such as scanning electron microscopy (SEM), energy dispersive X-Ray (EDX), X-Ray diffraction (XRD) and Raman spectroscopy. Results showed greater depth of neutralization and surface pH reduction in FA-GPm as compared to AASm. Further investigation on penetration of sulphur confirmed much higher diffusion in FA-GPm as compared to AASm. XRD and SEM with EDX analysis showed the crystallization of thenardite and gypsum within the matrix of FA-GPm and AAS mortar, respectively, which was also, confirmed by Raman spectroscopy results. Extend of overall matrix deterioration was much higher in FA-GPm as a result of thenardite crystallization as compared to AASm after 12 months of exposure.

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## 1. Introduction

Concrete is the most consumed construction material which is used in almost every project including roads, buildings, hydraulic structures, bridges, etc. Alkali-activated binders also known as

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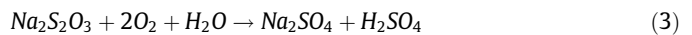
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'Geopolymers' are considered as a new trend in concrete technology and have been extensively investigated during the past decades. The primary difference between these materials and conventional ordinary Portland cement (OPC) is that they are free from clinker. Clinker is manufactured by burning limestone (calcium carbonate) and siliceous materials, causing a heavy production of CO<sub>2</sub> and an extensive consumption of energy, ranging from 4 to 5 GJ per ton of clinker [1], resulting in very high carbon footprint and energy requirements [2]. Geopolymer is a potential alternative in the field of sustainable infrastructure development. Geopolymer concrete is a synthetic material having a carbon footprint up to 40% lesser as compared to conventional OPC concrete [3]. They are produced by the reaction of solid precursors such as fly ash (FA), metakaolin and ground granulated blast-furnace slag (GGBS) with an alkali metal hydroxide or silicate solution. Depending on the precursor used, the final products can have considerably diverse nano/microstructures with different properties, varying the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> and CaO/Al<sub>2</sub>O<sub>3</sub> oxides ratios [4]. There are generally two different models on the basis of which these alkali-activated binders are made. First model consider lower CaO content in the original material, whereas the second model consider higher CaO/Al<sub>2</sub>O<sub>3</sub> ratio, generally more than 3 [5]. During the alkali activation of aluminosilicate-dominated metakaolin and FA having low CaO, a highly cross-linked alkali aluminosilicate network, also known as geopolymer [6,7] forms; with an intrinsically porous nano/microstructure [8]. Alkali activation of calcium-rich slag leads to the production of an amorphous alkali charge-balanced aluminium-substituted calcium silicate hydrate gel [9–13] that is similar to C-S-H in OPC concrete, with a finer pore structure than those of aluminosilicate networks. This alkali-activated slag in comparison to low calcium FA based geopolymer, have usually better mechanical properties lower heat of hydration and better response against chemicals, however, they have disadvantages like quick setting and higher shrinkage resulting in microcracking [14]. Since geopolymer shows similar mechanical properties to those of conventional OPC concretes [6,15–19], its long-term durability performance is yet to be determined [20–22], especially against aggressive biochemical environments like sewers etc.

In the area of wastewater management, structures for collection and transportation sewage are generally made from concrete, making it susceptible to deterioration. This degradation is a multistage process under highly acidic environment. The theory behind this degradation was first introduced by Olmstead and Hamlin (1900) [23], when the cement mortar lining was corroded in an outfall sewer within 5 years of construction. They assessed this deterioration of mortar lining and linked it with sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) resulting in the formation of expansive gypsum causing cracks and fracture. In 1945, Parker [24] also observed the degradation of concrete in sewage environment linking it with biochemical activity. Further, five strains of sulphur oxidising microorganisms (SOM) known as *Thiobacillus* were also isolated from the surface of concrete which was corroded as a result of their ability to oxidate sulphur into H<sub>2</sub>SO<sub>4</sub> in high sulphur environments [24], hence the term "Microbially Induced Concrete Corrosion" was introduced. This MICC results in the degradation of microstructure and performance failure reducing the life span of concrete from expected 100 years to almost 30–50 years and in an extreme cases to 10 years [25].

MICC is a multi-staged process, starting from sulphate reducing bacteria (SRB) e.g. *Desulfovibrio*, under anaerobic conditions at the bottom most of the pipe, to produce hydrogen sulphide (H<sub>2</sub>S) by reducing oxidized sulphur compounds. This H<sub>2</sub>S converts from liquid to gaseous form and escapes to the atmosphere as a result of turbulence and is the main source of H<sub>2</sub>SO<sub>4</sub> formation [26]. The deterioration of concrete takes place in an aerobic environment under the influence of sulphur oxidizing microorganisms (SOM)

which in high sulphur environment absorbs this sulphur and converts it in to H<sub>2</sub>SO<sub>4</sub> causing major microstructural deterioration of concrete. This mechanism is known as bacteriogenic or biogenic corrosion of concrete [27]. Thiosulphate (S<sub>2</sub>O<sub>3</sub><sup>2-</sup>) and elemental sulphur (S<sup>0</sup>) are among the several intermediates in the main oxidation of H<sub>2</sub>S to sulfate (SO<sub>4</sub><sup>2-</sup>), which acts as a substrate for many *Thiobacilli* SOM such as *Thiobacillus thiooxidans*, *Thiobacillus thio-parus*, *Thiobacillus intermedius*, and *Thiobacillus neapolitanus* [27]. The metabolic effect of *Thiobacillus* and neutralization of concrete increases with the increase in concentration of hydrogen sulphide, moisture and oxygen [28]. These organisms oxidize the H<sub>2</sub>S in the atmosphere into elemental sulphur and deposit it inside their cell [29–31]. As a result the environment gets depleted from H<sub>2</sub>S gas. So, in the deficiency of H<sub>2</sub>S they convert this elemental sulphur in to H<sub>2</sub>SO<sub>4</sub> [29], as shown in Eqs. (1) and (2). Similarly, thiosulphate also oxidizes into H<sub>2</sub>SO<sub>4</sub>, however that produces only one mole of H<sub>2</sub>SO<sub>4</sub>, making it less aggressive as compared to elemental sulphur, as shown in Eq. (3) [29].



The degradation process of concrete in sewer is shown in Fig. 1. The freshly placed concrete, depending upon the type of mix, is initially having a very high pH ranging from 12 to 13. Concrete is initially not favourable for the growth and colonisation of bacterial species. Islander et al. [30], proposed three staged deterioration of concrete in sewers, starting with an abiotic chemical reaction with CO<sub>2</sub> known as carbonation and acidification of H<sub>2</sub>S present in the air to thiosulphate and polythionic acid, causing degradation of concrete and reducing pH down to 9, especially on the surface [30]. These abiotic chemical reactions are followed by MICC on the surface as a result of microbial colonies growth. Neutrophilic sulphur-oxidizing microorganisms (NSOM) grows first at pH around 9.0 e.g. *T. Thioparus*, *T. novellus* etc., and utilizes the sulphur compounds emitted from the sewage, producing elemental sulphur and polythionic acid to further reduce the pH of the concrete from 9 to around 4 [32]. These NSOM continuously consume the sulphur compounds to acid and colonize the concrete till pH reaches 4–5 as it limits their growth, and ends the stage 2 of MICC. Afterwards, further reduction in pH is the result of acidophilic sulphur oxidizing microorganisms (ASOM) which replaces NSOM e.g. *T. Thiooxidans*, *T. intermedius* etc. These microorganisms further reduce the pH of concrete by metabolizing sulphur and thiosulphate into H<sub>2</sub>SO<sub>4</sub>, as shown in Eqs. (1)–(3) and can sustain pH level below 2 [33].

Since concrete is porous and microbial colonies in the environment are three dimensional, this corrosive environment provides enough microorganisms and compounds to distribute throughout the volume of the concrete [30]. Sulphates not only on the surface but also within the concrete react with water in the presence of microorganisms to produce H<sub>2</sub>SO<sub>4</sub> neutralizing alkalinity within the concrete. The attack of biogenic sulphuric acid results in the dissolution of cementitious phases. In case of OPC concrete, Portlandite (Ca(OH)<sub>2</sub>) and calcium silicate hydrate (C-S-H) losses Ca<sup>2+</sup> as a result of acid attack and forms gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O) which further forms ettringite (3CaO·Al<sub>2</sub>O<sub>3</sub>·3CaSO<sub>4</sub>·32H<sub>2</sub>O) as a result of reaction of gypsum with monosulfoaluminate or C<sub>3</sub>A [34,35]. The high performance of geopolymer against the H<sub>2</sub>SO<sub>4</sub> is established [19,36–38] but the deterioration reactions are neither straight forward nor similar in every kind of geopolymer. Acid attack on low calcium fly ash based geopolymer results in the replacement of alkali cation with H<sup>+</sup>, as shown in Eq. (4). However, when it is exposed to much more aggressive acid, the chains of aluminosili-

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