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Research Article

Study of Different Microbial Corrosion Mechanisms in Sewer Pipes Network Made by Sulfur Concrete with focus on Strength and Durability Analysis

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Abstract

Concrete corrosion has a great economic impact on many worldwide projects especially in corrosive environments such as municipal sewer systems. Many different factors can cause corrosive attacks on concrete structures that can be categorized into two main groups, chemical sulfuric acid attack, and microbial corrosive process via producing biogenic sulfuric acid (BSA). The present work aimed on assessing the sulfur concrete durability in sewerage environment and investigating the effects of microbial corrosion on this type of concrete. In this study, two tests were conducted including sewer concrete pipe in situ, and experimental tests on cubic samples. For biological tests, this bacterium was chosen for performing laboratory tests since the presence probability and population of *Thiobacillus thiooxidans* microorganism in domestic sewer is higher than other species. Results showed that sulfur concrete is considerably resistant to severe acid attack, but is less resistant to microbial corrosion attack than chemical corrosion especially against sulfur oxidizing bacteria. This is completely different in Sewer environment, because the microbial attack is limited by some factors such as pH variety and other bacteria presence competition.

Keywords: Sulfur concrete, Microbial Corrosion, laboratory experiments, Sewer Environment, *Thiobacillus thiooxidans microorganism.*

Introduction

Concrete corrosion has a great economic effect on many worldwide projects especially in corrosive environments such as municipal sewer system. Therefore, in these projects using suitable materials which are resistant to corrosive factors is a vital decision (Kasani and Hamidzadeh, 2018). Iran has many Sulfur providing potentials in both mineral or by product shape. Nowadays, almost of all elemental sulfur is made from removal of sulfur-comprising contaminants from petroleum and natural gas which can be considered as petroleum waste and also sulfur bio-leaching in mines. The greatest commercial use of this element is the production of sulfuric acid, phosphate and sulfate

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fertilizers, other chemical products, and sulfur concrete. Sulfur concrete has been known as innovative product with useful properties such as chemical corrosion resistance, high stability, long-term perdurability, and impermeability. A lot of research has been done in the world due to the importance of the durability of concrete sewer pipes by Fatima and Muntean in 2014. In 1930, Parker in Australia and, Pomeroy in the United States conducted extensive research to determine the corrosion rate and the estimated useful life of concrete sewer pipes. In 1970, Meyer and Ledbetter specifically examined the effect of concrete treatment in preventing corrosion of concrete pipes. In 1997, Daczko and Johnson investigated the role of polymer excessive materials in controlling the corrosion of sewage pipes in addition to the effect of different acidic environments. In 2004, De Belie et al. in a form of an experimental study examined and predicted the chemical and biological effect of sulfuric acid on various types of industrial-grade concrete sewage pipes. In order to estimate and predict the corrosion rate, they have investigated the effect of aggregates in concrete types and concluded that the type of aggregates used, if calcareous, exhibits higher resistance than silica and slag aggregates due to the type of compounds and also the porosity created to absorb water concrete corrosion caused by sewage is not a time linear process. Before there is visible corrosion, there is a primary period in which the microbial population decreases and increases and, the chemical and physical properties of concrete vary considerably. When the mass is lost, the corrosion rate does not remain linear because the environmental conditions (sewage chemistry, pH, temperature) and concrete properties (corrosion layer, microbial population, concrete chemistry and porosity) continue to change. This kind of concrete has many other advantages, such as achieving up to 90% of its ultimate compressive strength in a day, and no need to curing and water for its production (as cement concrete needs). All of these advantages make this kind of concrete a perfect candidate for installation in extreme corrosive circumstances like sewer system.

Many different factors can cause corrosive attack to concrete structures that can be categorized in two main groups: 1- Chemical sulfuric acid attack, 2- Microbial corrosive process via producing biogenic sulfuric acid (BSA). Some researchers attempted to simulate the corrosion procedures and investigate each of these factors in cement concrete corrosion. In Hamburg, a simulation chamber was built for microbiological corrosion tests in which specific conditions of temperature, nutrients and humidity can be controlled (Sand, 1997). The rate of corrosive BSA attack was determined by the test specimens' weight loss (cubes of $1.8 \times 1.8 \times 2$ cm) and water pH where the concrete blocks were submerged. Mori used also a simulation chamber and in their tests to determine the corrosion rate by measuring the reduction of the cross section of the mortar samples (Mori et al., 1992). Schmidt described another type of simulation reactor developed by the Heidelberger Zement AG. Samples were removed from the reactor at monthly intervals in order to determine the cell density at the surface as well as the loss in weight due to corrosion (Schmidt, 1997). They figured out that the main source for biogenic sulfuric acid is the sulfur compound hydrogen sulfide, H_2S. It is made by sulfate-reducing bacteria (SRB, such as *Desulfovibrio* sp.). The second is active under anaerobic circumstances and reduces oxidized sulfur compounds to H_2S. These microorganisms are responsible for H_2S formation, while they are live in the sewage, mud at the pipelines' bottom, and slime layer (biofilm) covering the pipelines surfaces below and above the water level. H_2S is transformed into sulfuric acid after the H_2S sorption from the sewer environment into the biofilm or concrete the on the pipelines' surface above the water line under aerobic circumstances. When the H_2S reaches the atmosphere, it may have reaction with oxygen as elemental sulfur deposited over the slime layer, covering the walls. The aggressive produced sulfuric acid may attack the concrete pipe's inner surface and other parts of the transportation and treatment facilities like pumping stations, reservoirs, and manholes. The resultant corrosion products such as ettringite and gypsum can be created (Bock et al., 1988; Grengg et al., 2019).

These expansive products can result in the incremented internal pressure leading to small cracks. Besides, the corroded materials can be eliminated by the sewage flow also causing the corrosion acceleration (Lei and Itao, 2018). This work aimed to assess the sulfur concrete durability instead of Cement concrete in sewerage and investigating the effects of microbial corrosion on this type of concrete.

Materials and Methods

Concrete specimens

In this work, two types of experiments were conducted including laboratory experiments on cubic samples and in situ tests on sewer concrete pipe. It was aimed to assess the sulfur concrete's microbial corrosion in the worst case and controlled conditions in the laboratory scale. In this regard, the natural microbial corrosion was observed in a real situation in sewer treatment plant. Sulfur Concrete specimens were made via a mix design containing 70% standard sand, 25% elemental sulfur and 5% SMZ additive based on ACI 548.2R-93 (ACI, 1993). Through preheating the solid aggregates in 120-160°C, then, they were mixed with additive (sulfur cement) and melted sulfur in a mixer at 120-140 °C until obtaining a mixture homogeneous substantially. Then, the created mixture was cast and formed into cubic mold (5 cm × 5 cm × 5 cm) and pipe mold (length of 100 cm, diameter of 15 cm, and thickness of 3 cm) (Xie et al., 2018).

Samples installation

Pipe samples were mounted in sewer entrance canal of Tehran Shahid Mahalati Wastewater Treatment Plant (Tehran, Iran) after providing the sulfur concrete specimens. The plant possesses online monitoring systems to measure sewer quality parameters like temperature, BOD, pH, TDS, and COD (Li et al., 2017; Pacheco-Torgal et al., 2015). One of the pipes was eliminated each 3 months to measure the material's corrosion reaction. The tests were sustained up to a year to obtain the correlated data. Some laboratory tests were also performed with cubic specimens. The cubic specimens were located in a biological growth chamber containing a glass box (20 cm× 10 cm × 40 cm) for accommodating 12 sulfur concrete cubic specimens (5 cm × 5 cm × 5 cm) within an incubator. The location of cubic sample in this pilot is represented in Figure 1. Two samples were eliminated each month for test analysis.

Culture media and bacteria selection

Sewage includes various bacteria, which can be occasionally a corrosion operator by material degradation or formation of biogenic acid. Sulfur is a substrate for several thiobacilli bacteria like *Thiobacillus neapolitanus*, *Thiobacillus thiooxidans*, and *Thiobacillus intermedius* (Kelly et al. 1997) metabolizing it into sulfuric acid. Clearly, sulfuric acid has no severe corrosion impacts on sulfur concrete regardless of cement concrete. However, it is essential to the metabolism process (Vincke et al. 2002).

Because *Thiobacillus thiooxidans* microorganism has higher presence probability and population of in domestic sewer than other species, they were chosen to perform experimental tests. The bacteria seeds were achieved from microorganism bank of Iranian Biological Resource Center

(IBRC). For propagating bacteria, the culture media was created in terms of IBRC instructions. To culture the bacteria, 200 mL flasks were used comprising mineral salts solution (100 mL), which was supplemented with sulfur as the energy source Gu et al. (1998). Ensuring the propagation procedure and presence of the bacteria in culture media (Grengg, et al. 2018), some microscope observations were conducted. Incubation of the flasks was performed for 2 weeks on a rotary shaker at 2 ± 28°C. For these bacteria, the favored pH growth range was around 0.5 to 3. The preparation steps are displayed in Figure 2.

Figure 1. Biological pilot

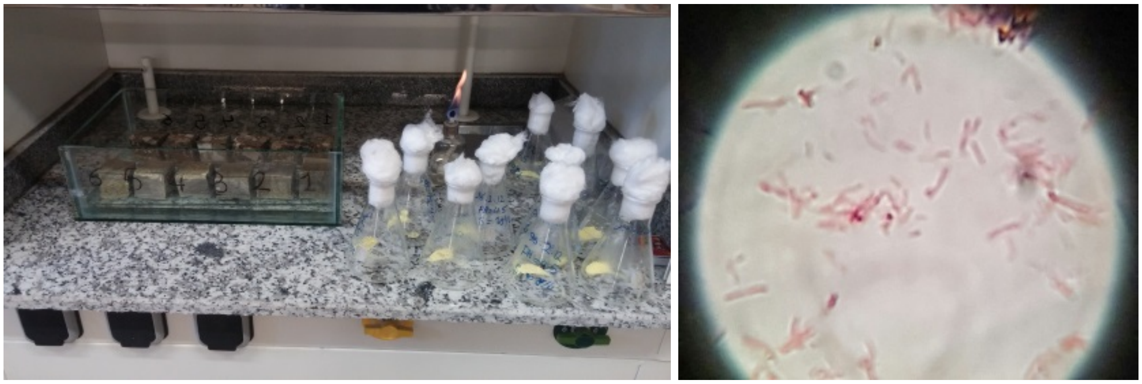


Figure 2. The incubation process and culture media

Biological Experiments

By measuring PH value during biological test and after exposure of sulfur concrete to concentrated bacteria culture, the bacteria activity was recognized and their concentration was discovered through plate counting. Nutrient solution was also added (designed for supporting the culture media and the bio reactor), if needed (Grengg et al., 2015). The reactor was designed with suitable open surface for aerating during incubation appropriately by maintenance of adequate oxygen (Santo Domingo et al., 2011). Severe microbial corrosion condition was provided by adding fresh sterile growth medium every 2 weeks (Ding et al., 2017).

Determining the sulfur compound in sulfur concrete

To discuss the effects of bacteria on concrete corrosion further, the shape of sulfur in sulfur concrete surface must be understood. Hence, X-ray diffraction (XRD) and X-ray fluorescence (XRF) were used. By XRF test, a sample's chemical parts are defined by measurement of the reflected secondary X-ray and excitement via a main source of X-ray. XRD is a nondestructive and compatible analytical method representing comprehensive chemical and structural data on the materials' crystallography. A sample's elemental content is determined through XRF analysis. However, it provides no information on the integration of different elements together. The results of XRD and XRF analyses on concrete samples revealed that in sulfur concrete more than 90% of sulfur was not in definite compound and it was found as elemental sulfur. Determination of sample mass loss is a main analysis to quantitatively describe concrete corrosion. Hence, the untreated samples' weight was determined at the start of the tests utilizing a 0.1 mg-accuracy digital scale (Huber et al., 2017). Through the tests, the specimens were eliminated from the reactors and washed to eliminate residual solutions. Drying the specimens at 40 °C until weight constancy (Jia et al., 2019), they were reweighted while recording the difference in weights.

Determining the of concrete samples' mechanical features

Determination of compressive strength was based on ASTM C109/C109M (ASTM 2016). In various settling times, the 5 cm × 5 cm × 5 cm cubes were treated in the reactor and eliminated from the pilot. Using a compression machine (Forney, Zelienople, Pennsylvania), the test was performed. At P=60-180 KN, the cubes were loaded until reaching the maximum load. To calculate the peak stress (fc), by the ultimate load at peak (P) was divided by the initial average cross-sectional area (A) as follows:

$$fc=\frac{P}{A} \tag{1}$$

ICP-OES, SEM, EDX

Determining the concrete corrosion extent, the corroded elements in the reactor solution were analyzed with Inductively Coupled Plasma spectrometry (ICP-OES). By this technique, the alterations in concrete composition were characterized. Moreover, the elements leaching out of specimens and corrosion products were identified. Moreover, element precipitation within solution reactor represent the concrete corrosion process.

The ICP-OES tool VISTA-PRO model with feed rate of 1.4 ml/min, CCD detector, and Plasma flow of 15 l/min Concentric Nebulizer was chosen. Another way to detect concrete surface corrosion and observe the microbial effects on Hole Index Ratio (HIR) is to use Scanning Electron Microscopy (SEM), SEM-MAP and Energy Dispersive X-ray spectroscopy (EDX), MAP and EDS analysis for further obtaining information on element distribution and microstructure on concrete surface. Thus, the samples' surfaces were covered with a thin gold layer for avoiding charging. The SEM was (VEGA3-TESCAN, Czech Republic) used at an accelerating voltage of 20 kV. Discovering the distribution of the elements over the exhibited area was conducted via a detector (RMRC RONTEC- MAP, Germany).

Results and discussion

Biogenic acid effect and microbial activity

Sulfur degradation process and aerobic microbial oxidation of hydrogen sulfide into biogenic sulfuric acid are complex and may include various stages. Figure 3 shows a simplified sketch trend towards various oxidized types of sulfur (Islander et al., 1991; Li et al., 2018).

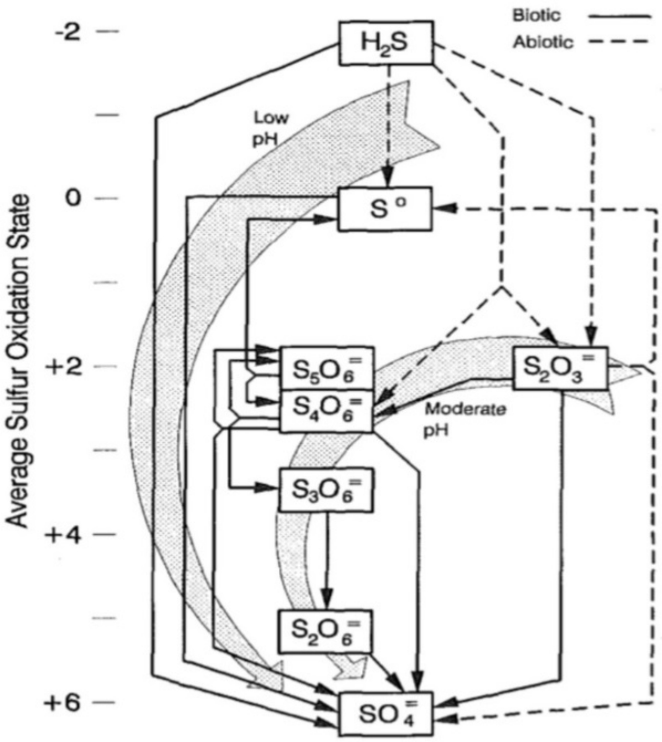


Figure 3. Pathways for sulfide oxidation

Some aerobic community of microorganisms at the top surface of concrete can oxidize the sulfide to corrosive sulfuric acid. A complex microbial ecosystem, which occurs in sewage environment, has the ability of generating sulfuric acid which causes corrosion of concrete. Initially highly alkalinity of cement concrete surface resist against acidic changes on it (Bertron, 2014; Usher et al., 2014). In a natural ecosystem microbial succession occurs and based on some environmental factors like pH the surface is dominated by some species. After passing time, microbial activities on concrete surface provide a conduit for ions that go through the surface. So, after sulfide oxidation, sulfuric acid diffuses and can neutralize the protective alkaline characteristic of concrete. Some chemical reactions that occur at the same time increase the porosity of concrete (Sun et al., 2016).

The sulfide oxidizing microorganism's microenvironment on the concrete can be changed by precipitation of some chemicals. Low pHs causes the domination of acidophilic microorganisms. In addition, using sulfide as a single energy source makes it more probable. It is possible to produce and use elemental sulfur in the treated Sulfur Concrete surfaces reactions at lower pH. This type of reaction occurs rapidly through thiosulfate oxidation by a pH of 4 (Harrison et al, 1984; Song et al., 2019; Santo Domingo et al., 2011).

The corroded pipes crowns often exhibit yellow deposits. Seemingly, elemental sulfur was utilized by some species as the favored or even the only sulfur substrate. Hutchinson concluded the growth of Thiobacillus *thiooxidans* at less pH values in comparison to any other substrates (Hutchinson et al., 1969). It was found that this microbial activity reached its maximum level by achieving the worst case, however, pH was kept at lower ranges under experimental circumstances. Sulfide oxidation leads to 8 electrons (23.8 kcal/mole per sulfide equivalent). Four electrons (29.9 kcal/mole per carbon equivalent) were obtained by carbohydrate oxidation (Stump et al., 1992). Therefore, sulfide input energy was 2.6×10^{-4} moles of carbohydrate/m^2/hr. Therefore, the organics excreted by acid-generating bacteria was exposed to degradation owing to a mutualistic association with acidophilic heterotrophs needed for the constant fast growth. In this work, BSA was produced by *Thiobacillus thiooxidans* affecting CC. However, SC corrosion sulfur metabolization in the second case requires further studies (Onet, et al., 2018).

More possibly, acid formation by these organisms was self-inhibitory owing to the pH limitation. The experimental observations on the solution pH values in the reactor supported this probability. However, *Thiobacillus thiooxidans* growth can be reduced considerably in the natural sewer canals as a result of the existence of a higher range of pH (5-8). It was concluded that the most considerable cause of SC microbial corrosion was elemental sulfur oxidation under accessible preferred circumstances. In this survey also the activity of acid producing gram negative rod shape bacterium, *Thaiobacilus thiooxidasns* was specifically investigated on the sulfur concrete surface in a batch mode experiment design. This species can oxidize sulfur and thiosulfate, reducing pH to less than 3 (Huber et al., 2017). The bacterium is an obligate autotroph that grows well to pH 1 (Mildeetal 1983). As it has been mentioned in *Bergey's Manual*," it has a remarkable ability to oxidize elemental sulfur rapidly".

As sand and Bock has reported *T.thiooxdans* doesn't form a slime layer or biofilm on concrete[5]. So, our observations based on SEM micrograph pictures haven't shown biofilm any way. Although determination of the concrete corrosion rate extent based on ICP-OES analyzes showed characterized alterations in concrete composition and concrete corrosion procedure on bacterial aggregations on concrete was observed through SEM micrographs. There are many environmental factors which can limit bacteria growth in a field condition. Low pHs, nitrogen and carbon dioxide are the most important of them. pH decreased through bacteria activities in experiment chamber and it was about 2 during the time that could really cause low growth rate.

For the samples which were kept in suit (in sewer) biological activities of other organism's leads to producing large amount of CO_2 and carbonates are also released by concrete corrosion. Nitrogen is another essential growth element. Biological acid producing may eliminate fixed nitrogen from concrete surface although ammonia is a part of sewer atmosphere and could be used as a nitrogen source. In batch experiments nitrogen was provided in a low amount as $(NH_4)_2SO_4$ (part of culture media) and carbon dioxide was provided from atmosphere.

A comparison between surface characteristics of Sulfur concrete before and after exposing to *T.thiooxidans* based on SEM micrographs showed bacterial activity and sulfur oxidizing that leaded to surface changes but no biofilm or slime layer formation (Ilie et al., 2018). About the concrete samples placed in contact with real sewage produced biological acid may wash away or neutralize by sewage components.

Visual examination of the specimens

Treating the samples in laboratory reactor and sewer canal, a comparison between the untreated and treated samples elaborates the difference of observed degradations. Mostly no degradation was observed in field samples but in laboratory samples a thin layer of salt crystals was found on the concrete surface. This layer concentrations shows that the HIR factor seemed to be more for treated samples in comparison with untreated ones.

Figure 4 represents a treated sample's relative mass to its mass amount before test as a function of exposure time. The samples that showed the most extreme behavior, in terms of mass loss, were those that provide better conditions for microbial activity in longer time than others. The corrosion circumstances within the parallel set-up were comparable for more confidence. Increasing the weight slightly at the start of the experiment is caused by densification of the concrete matrix and salt crystal sedimentation.

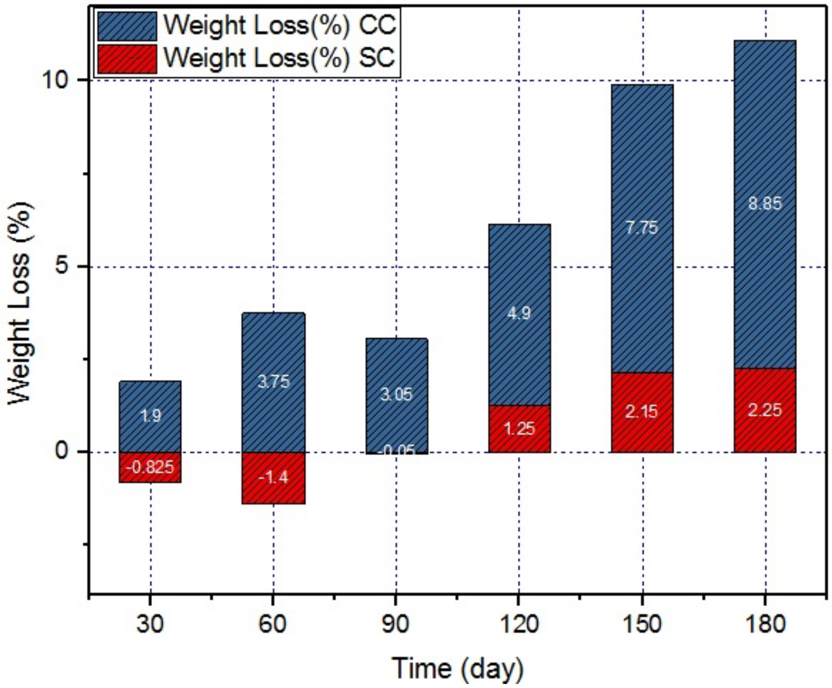


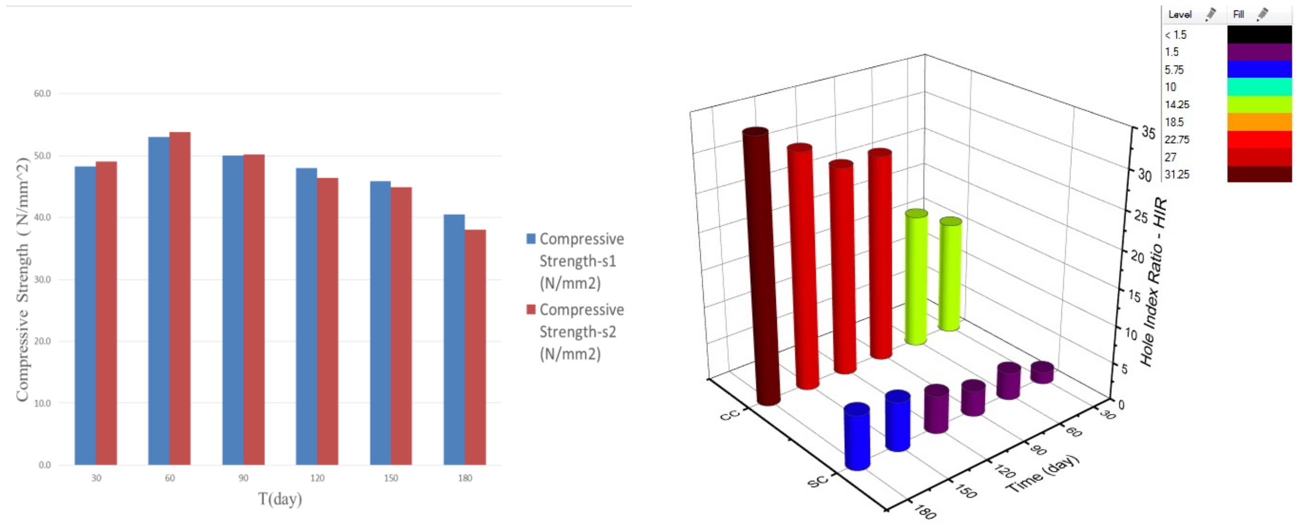
Figure 4. Samples' weight loss in biologic reactor

Compressive test discussion

Figure 5a shows time-based alterations in compressive strength. It is believed that the reduced compressive strength was attributed to cracking within the samples (Asamoto et al., 2011). Increasing the HIR factor also contributed to the decreased compressive strength (Figure 5b). Indeed, these factors both led to crack expansion and tension concentration. Thus, the sampled are permitted for constant penetration of bacteria colony and improving the damaging cycle. The alterations in compressive strength in these tests were related considerably to HIR factor.

Reactor solution ICP test discussion

As stated in sulfur concrete corrosion process, the sulfur polymer detoration can precipitate some aggregate elements in solution reactor that indicates the element distribution and process analysis in concrete surface. According to Fig. 6, calcium, sulfur, and siliceous elements concentrations in solution are incrementing as exposure time increases.



(a) Compressive strength of samples (b) HIR factor during exposure time

Figure 5. Compressive strength changes and HIR factor

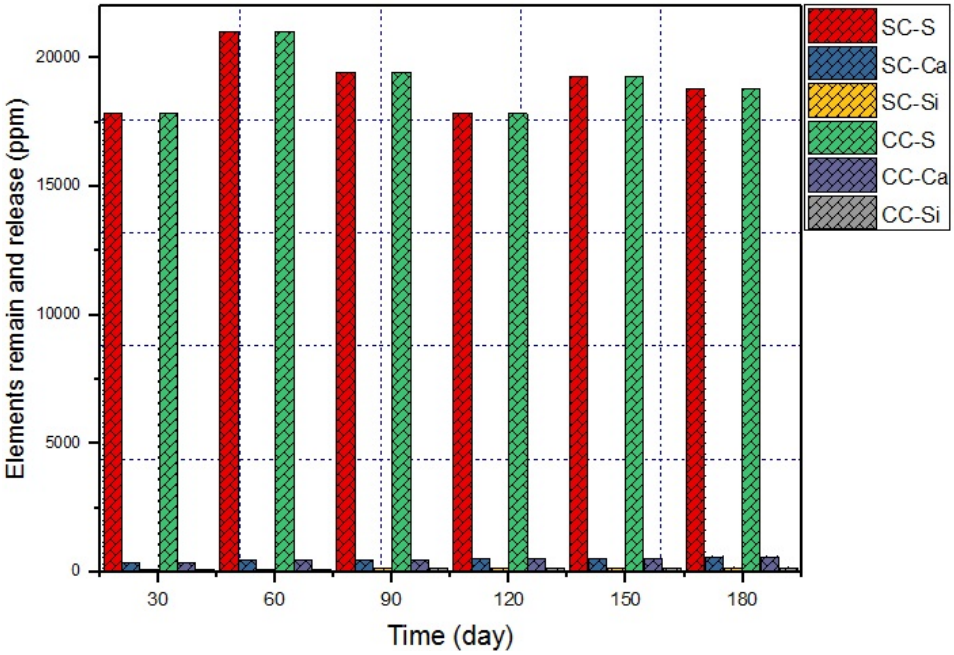
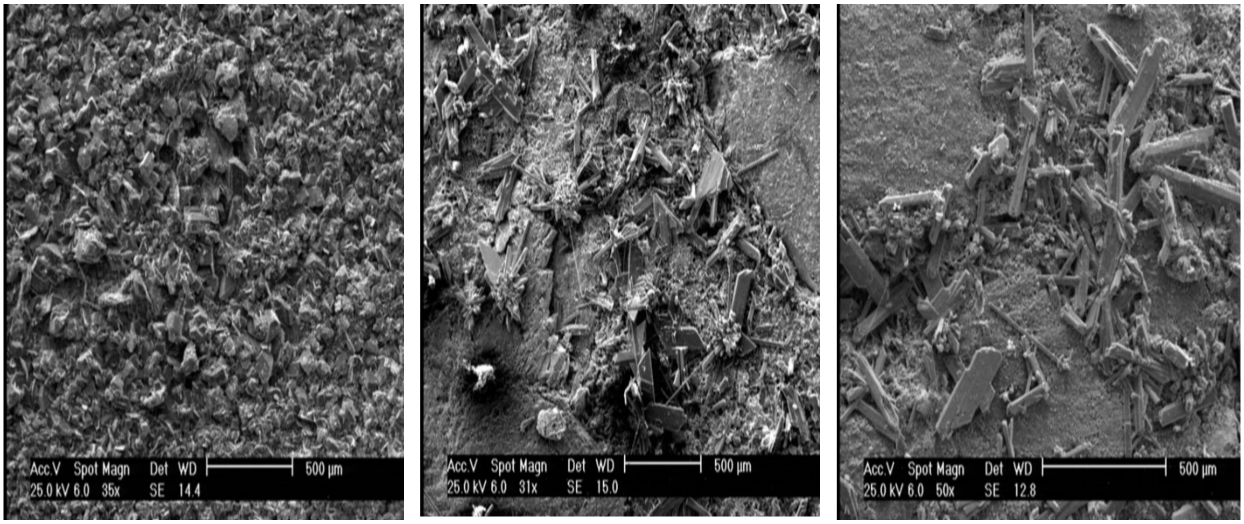


Figure 6. ICP detection for leached elements

Results of SEM

The results of SEM (Figure 7) reveal that the sulfur concrete matrixes mainly comprise sulfur-coated materials (fillers and aggregates), in which, sulfur was accumulated in the voids within the particles. According to the XRD results, before and after the exposure, the SC major mineral phases were stable. SC was resistant to biogenic and mineral acids and higher salt atmospheres (McBee and Weber, 1990), but it has lower resistance to severe microbial attack.



(a) SEM analysis of an untreated sample

(b) A sample exposed for 6 months in the biologic reactor

(c) A sample exposed for 30 days in the chemical reactor

Figure 7. SEM results for laboratory samples

Furthermore, the SEM analysis showed the incidence of bacteria in concrete holes (Figure 8) and in these locations elements concentration in MAP analysis (Figure 9) clearly represented the aggregate outcast and sulfur degradation. By setting these consequences with ICP analysis, the microbial corrosion in sulfur concrete is justified clearly. The main objective of the study of SEM images in evaluating samples tested in a biological reactor is to detect the presence of bacteria and the type of holes caused by microbial activity in each of the concretes.

Conclusion

After conducting the tests and analyzes on the environmental factors of sewage on the corrosion of sewage pipes made of sulfur concrete in comparison with cement concrete (ordinary concrete), it can be concluded that the main cause of the destruction of pipes made of cement concrete, in the simulated or laboratory sewage environment, chemical corrosion is the result of the reactivity of sulfuric acid condensed in sewage gases or biogenic acid derived from microbial activity by sulfur oxidizing bacteria. The process is that by dissolving the cemented polymer and transforming it into etringite and gypsum, the corrosion of concrete samples and pipe crown occur. Field and laboratory investigation of sulfur concrete in corrosive environment can provide a good situation for studying the corrosion factors in microstructure scale. In the experiment chamber pH changes was detected every 2 hours a day after adding fresh culture medium for 5 days. Decreasing in pH value was evident during 3 first days after adding fresh culture medium. A decreasing in pH value combined with the increased sulfate concentration in the effluent indicates the bacterial activity and sulfuric acid production [12] which both of them was observed during the experiment time. The pH value of culture medium after 10 days of cultivation of bacterium was about 3.5 and after 52 hours of adding to experimental chamber reached to 2.8 showed the bacterial activity to oxidizing sulfur as a sole energy source. pH decreasing continued during 3 months later but because of dissolution of concrete, which releasing alkaline products it was not very quick or steady. Monitoring sulfate changes in batch experiments is highly recommended in order to observing bacterial activity.

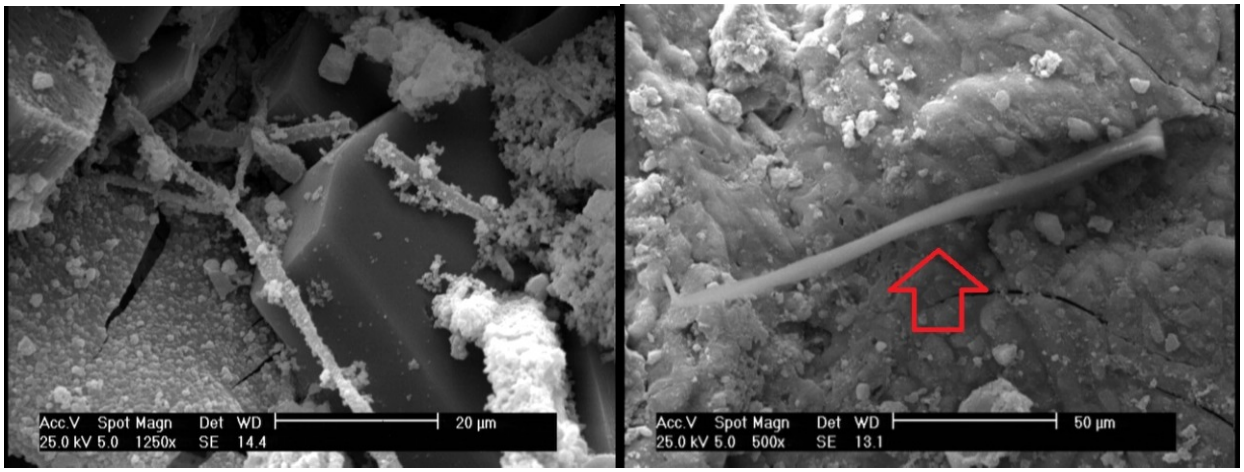
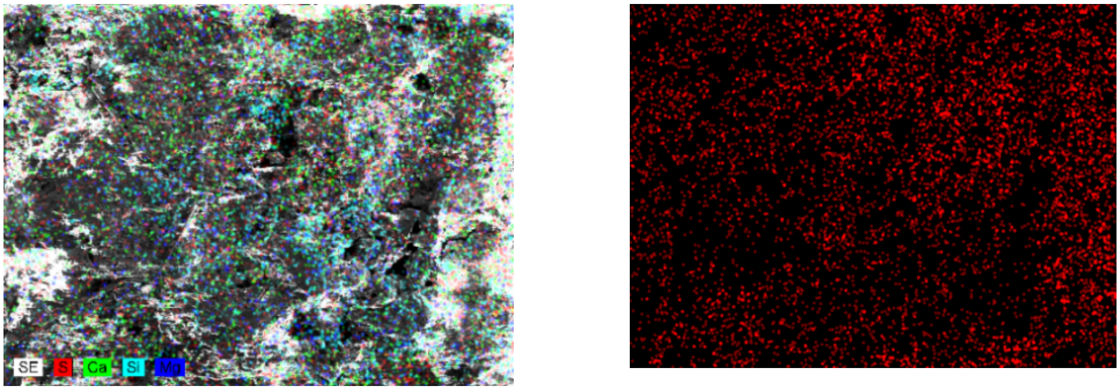
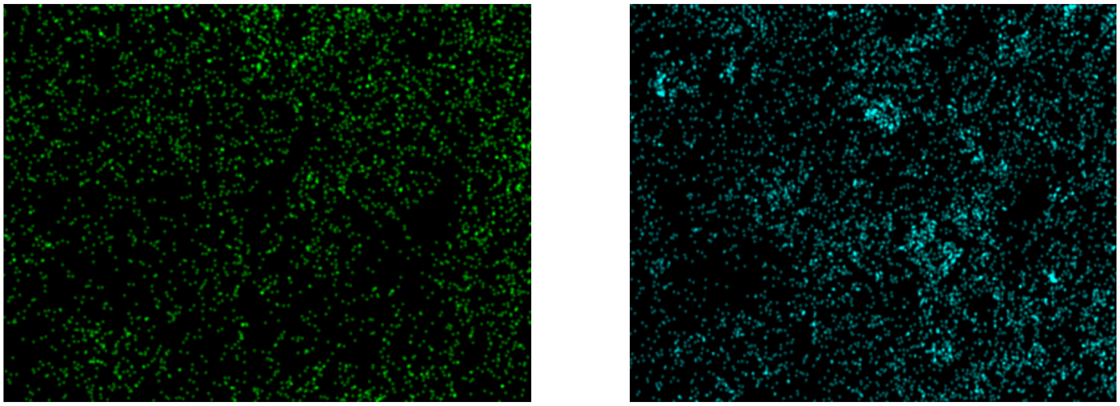


Figure 8. Bacteria detection in SEM analysis



General MAP

Sulfur distribution



Calcium distribution

Silica distribution

Figure 9. MAP Analysis for S, Ca, and Si

It is concluded that sulfur concrete is considerably resistant to severe acid attack but is less resistant to microbial corrosion attack than chemical corrosion especially against sulfur oxidizing bacteria. In sewer environment, this condition is completely different due to microbial attack limitations by some factors such as pH variations and other bacteria present competition. This study also indicates that sulfur concrete material has features that in definite applications are greater than cement concrete. SC as a durable material can be successfully used in sewerage networks in semi-arid and arid areas. It has several benefits over cement concrete when located in a sewerage setting.

Sulfur concrete is not influenced by sulfuric acid or hydrogen sulfide made in sewer systems. It is a high-strength, acid-and salt -resistant, and impermeable concrete suitable for using in strictly aggressive settings. Using sulfur concrete material in large-scale economical and provides a long-term durable solution for concrete deterioration problems. Moreover, a considerable amount of sulfur waste was made from the oil-refining procedure. Utilizing this sulfur in Sulfur concrete production, a considerable environmental problem related to the storage of this by product is solved. The use of sulfur concrete in sewage transmission networks increases the service life of the network by 60%, reduces implementation costs and time by 47%. Also, the use of sulfur in concrete is very effective in reducing the volume of waste depots for 20% by weight of sulfur consumption.

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