Investigating the effect of sewage corrosive agents on surface corrosion and porosity of cement concrete and sulphur concrete structures

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Abstract: The corrosion phenomenon causes an enormous economic and ecological damage every year around the world. The purpose of this study was to conduct experiments to investigate and accurately determine the effect of sewage environment on the behaviour of sulphur and conventional concrete as the main material of sewage network structures. For evaluating the corrosion behaviour of concrete samples, porosity factor was measured. By analysing the SEM, EDX, EDS images, a non-dimensional index called Hole Index Ratio (HIR) was determined. It can be concluded that the main cause of the destruction of pipes made of cement concrete is chemical corrosion while sulphur concrete is more resistant in this case but has lower vulnerability to biological corrosion. It should be noted that under the real conditions and the presence of microbial competition between bacteria and the unfavourable conditions for the *Thiobacillus thiooxidans* bacteria, this malignant effect is far less and is negligible.

Keywords: concrete corrosion; sewage network; hole index ratio; sulphur concrete; cement concrete; biological corrosion; chemical corrosion; *Thiobacillus thiooxidans*; microbial competition; scanning electron microscopy; SEM.

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1 Introduction

The corrosion phenomenon causes an enormous economic and ecological damage over billions of dollars every year around the world. This phenomenon has been abundantly observed in corrosive environments, including sewage and refinery networks and, causes the destruction of concrete structures and consequently sewage leakage, groundwater contamination, damage to process units and reduces the efficiency of the sewage treatment plant (Asamoto et al., 2011).

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 (Bertron, 2014). The nonlinearity of the corrosion process is an important problem for a researcher who is trying to predict the remaining life of the structures used in the sewage network. Therefore, it is appropriate to develop a more precise model for the corrosion process; a model that can estimate the instantaneous corrosion rate as a function of time during the lifetime of the sewage structure. The main focus of this research is to evaluate and accurately measure the behaviour of cement concrete and sulphur concrete specimens against biological and chemical corrosion to obtain the actual effect of these corrosions on each concrete specimen in the long term and in the actual sewage conditions by accurately analysing the results thus preventive measures or replacement of consumables can be put in the instruction. To achieve this goal, the development of a mathematical model for accurate prediction of corrosion with the highest compliance with real conditions and the least square error is one of the main requirements of the research and the definition of measurable parameters and accurate corrosion indicators are of high priority (Sabour and Movahed, 2017).

A lot of research has been done in the world due to the importance of the durability of concrete sewer pipes (Fatima and Muntean, 2014). In Australia and Pomeroy in the US

conducted extensive research to determine the corrosion rate and the estimated useful life of concrete sewer pipes. In another study, researchers specifically examined the effect of concrete treatment in preventing corrosion of concrete pipes (Kelly et al., 1997). In 1997, researchers investigated the role of polymer excessive materials in controlling the corrosion of sewage pipes in addition to the effect of different acidic environments (Sand, 1997). In 2015, Grengg et al. in a form of an experimental study examined and predicted the chemical and biological effect of sulphuric 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 (Grengg et al., 2015).

In this study, the introduction of indicators such as alkalinity and water absorption as a function of porosity were considered as the most important achievements of this research. In 2007, researchers evaluated the effect of sulphur oxidising bacteria on concrete corrosion in the sewage network. After accurate examination of the microbiology of corrosion, they pointed to the penetration depth of oxygen and H₂S into the concrete and considered the production of sulphuric acid by A. Thiooxidans as the main factor of concrete corrosion and reaction with the underlying layers. Another researcher examined the performance of conventional concrete with sulphur additive in sulphur environments and concluded that concrete containing sulphur should be made to provide a thicker structural function, while its corrosion resistance increases. Also, many studies determined the concrete corrosion model in sewage treatment and with different concrete specimens they conducted an experiment with six sewage samples for 48 months in Australia (Kasani and Hamidzadeh, 2018). The results of the model and their observations indicate that the optimal model was chosen for this process (Islander et al., 1991). Huber et al. in 2017 evaluated the degree of concrete corrosion after short-term and long-term contact with chemical and biological sulphuric acid. In addition to introducing the methods used, they concluded that there was no linear relationship between the two types of chemical and biological corrosion. In all cases, gypsum was formed as a corrosion product and there was no microbial growth in the corroded layer. Ding et al. also referred to the effect of concrete compounds on the resistance to microbial corrosion in 2017. They evaluated the effect of mortars made of calcium aluminate cement (CAC) and four sodium amine (SQA) against microbial corrosion at three weak, moderate and severe levels. The results of their research showed that, they show a good inhibitory effect on bacterial growth in weak and moderate corrosion of CAC and SQA. While inhibition in severe corrosion (pH ≤ 2), especially in relation to A. thiooxidans bacteria was negligible (Kasani and Hamidzadeh, 2018).

The corrosion of conventional concrete (cement) and its effective factors have been studied in most studies however, the interaction between various chemical and biological parameters, the comparison of the strength of different types of concrete against corrosion agents, especially in the sewage environment, has not been studied accurately. The purpose of this study was to conduct experiments to investigate and accurately determine the effect of different parameters and sewage environment on the behaviour of sulphur concrete and conventional concrete as the constructor material of sewage structures and to compare the behaviour of these types of concrete.

2 Materials and methods

Two kinds of experiments were performed for this study:

- 1 in situ experiments on sewer concrete pipe
- 2 laboratory experiments on cubic specimens.

This decision was made in order to study the microbial corrosion of sulphur concrete in the worst case and controlled condition in laboratory scale, while observing the natural microbial corrosion in a real situation within sewer treatment plant.

2.1 Concrete samples

For the exposure tests, CC samples were prepared by mixing cement, sand, and water at a weight ratio of 2:6:1.

SC specimens were produced through a mix design method incorporating standard sand (70%), elemental sulphur (25%), and SMZ additive (5%) according to ACI 548.2R-93.

Solid aggregates were preheated at 120°C–160°C and then mixed with melted sulphur and sulphur cement as an additive in a mixer at 120°C–140°C until a substantially homogeneous mixture was achieved.

The produced mixture was subsequently cast and shaped into a pipe mould with a length, diameter, and thickness of 100, 15, and 3 cm and cubic mould of $5 \times 5 \times 5$ cm, respectively. Figure 1 shows the specimens, as well as the casting procedure.

2.2 Samples installation

Providing sulphur concrete samples, pipe specimens were installed in sewer entrance canal of Shahid Mahalati wastewater treatment plant. This plant has online monitoring systems for measuring sewer quality parameters such as pH, temperature, BOD, COD, and TDS. Figure 1 shows the pipes locations in sewer canal. Each three months, one of the pipes was removed and corrosion reaction of the material was measured (Mori et al., 1992). The experiments were continued up to 12 months and the related data were obtained.

Figure 1 Samples installation (see online version for colours)



A series of laboratory experiments were conducted with cubic samples, too. The cubic samples were placed in a biological growth chamber consisted of a glass box (40 cm \times 20 cm \times 10 cm) which was designed to accommodate 12 sulphur concrete cubic samples (5 \times 5 \times 5 cm) and inserted into an incubator. Figure 2 shows the cubic sample location in this pilot. Each month, two specimens were removed for test analysis.



Figure 2 Biological pilot (see online version for colours)

2.3 Bacteria selection and culture media

Sewage has a large variety of bacteria, which some of them can be consider as corrosion operator by producing biogenic acid or material degradation. Sulphur is a substrate for many thiobacilli bacteria, such as *Thiobacillus thiooxidans*, *Thiobacillus neapolitanus*, *Thiobacillus intermedius* (Vincke et al., 2002), which metabolise it into sulphuric acid. Obviously, sulphuric acid does not have severe corrosion impact on sulphur concrete despite of cement concrete, but the metabolism procedure must be studied.

Since the population and presence probability of *Thiobacillus thiooxidans* micro-organism in domestic sewer is more than other species, these bacteria were selected for performing laboratory experiments. The bacteria seed was obtained from micro-organism bank of Iranian Biological Resource Center (IBRC). The culture media for propagating bacteria was made based on IBRC instructions. The bacteria were cultured in 200 mL flasks containing 100 mL mineral salts solution supplemented with sulphur as the energy source (Gu et al., 1998). Ensuring the bacteria were adapted to culture media and propagation process, some microscope observations were performed. The flasks were incubated on a rotary shaker at $28^{\circ}C \pm 2^{\circ}C$ for two weeks. The preferred pH growth range for these bacteria were about 0.5 to 3. Figure 3 shows the preparation steps.

Figure 3 Culture media and incubation process (see online version for colours)



2.4 Biological experiments

During biological experiments and after sulphur concrete exposure to concentrated bacteria culture, pH value was measured for recognising the bacteria activity and their concentration were explored via plate counting. Also nutrient solution (designed to support the bio reactor and the culture media) were added, if it was necessary. The reactor was designed with adequate open surface in order to aerate during incubation properly through maintaining sufficient oxygen. Fresh sterile growth medium was added every two weeks to provide severe microbial corrosion condition (Ding et al., 2017). Figure 4 shows the experiment condition.

Figure 4 Biological experiments (see online version for colours)

2.5 Sample analysis

2.5.1 Determination of sulphur compound in sulphur concrete

In order to go further in discussion concerning the effect of bacteria on concrete corrosion, it is necessary to know the sulphur shape in sulphur concrete surface. For this purpose, X-ray fluorescence (XRF) and X-ray diffraction (XRD) were utilised. XRF test defines the chemical parts of a sample by measuring the secondary X-ray reflected from it, when it is excited by a primary X-ray source. XRD is a compatible and non-destructive analytical technique that shows detailed structural and chemical information about the crystallography of materials. The elemental content of a sample is defined via XRF analysis, but it does not provide information about how the various elements are combined together. After analysing concrete samples with XRF and XRD, the results showed that more than 90% of sulphur in sulphur concrete was not in specific compound and it was observed as elemental sulphur.

2.5.2 Determination of mass loss

One of the main analyses for quantitatively description of concrete corrosion is determination of sample mass loss. For this purpose, the weights of untreated samples were measured at the beginning of the experiments using a digital scale with an accuracy of 0.1 mg (Huber et al., 2017). Conducting the experiments, the samples were removed from the reactors and rinsed in order to remove residual solutions. Then, the samples were dried at 40°C until weight constancy. Afterwards, the samples were weight out again and the difference in weight was documented.

2.5.3 Determination of mechanical properties of concrete samples

Compressive strength determination was performed in accordance with ASTM C109/C109M. The $5 \times 5 \times 5$ cm cubes were treated in the reactor in different settling times and removed from the pilot. A compression machine (Forney, Zelienople, Pennsylvania) was used to perform the test (Yin et al., 2018). The cubes were loaded at P = 60-180 KN until the maximum load was reached. Out of this test, the peak stress (*FC*) was calculated by dividing the ultimate load at peak (*P*) by the average initial cross-sectional area (*A*) using the following equation:

$$FC = \frac{P}{A}$$

2.5.4 Inductively coupled plasma spectrometry

Determining the extent of concrete corrosion, the reactor solution contained corroded elements were analysed with inductively coupled plasma spectrometry (ICP-OES). This method can help to:

- 1 Characterise changes in concrete composition.
- 2 Identify the corrosion products and elements leaching out of samples.

Furthermore, element precipitation in solution reactor is indicators for the concrete corrosion procedure. The ICP-OES instrument VISTA-PRO model, with CCD detector, concentric nebuliser, plasma flow of 15 L/min and feed rate of 1.4 mL/min was selected.

Figure 5 HIR detection procedure for porosity estimation, (a) SEM image before sample treatment (500 μm) (b)SEM image after sample treatment (500 μm) (see online version for colours)



2.5.5 Scanning electron microscopy, SEM-MAP and energy dispersive X-ray spectroscopy (EDX)

Another way for corrosion detection of concrete surface and observing the microbial effect on hole index ratio (HIR) is using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), EDS and MAP analysis to obtain further

information on microstructure and element distribution on concrete surface. Therefore, the specimen surfaces were coated with a thin layer of gold to avoid charging. The scanning electron microscope was (VEGA3-TESCAN, Czech Republic) at an accelerating voltage of 20 kV. Detecting elements distribution over the displayed area was performed by RMRC RONTEC- MAP detector, Germany. By analysing the SEM, EDX, EDS images, a non-dimensional index called HIR was determined by matching the images before and after the experiments and the empty space surface ratio to the initial level (Figure 5).

2.6 Paired comparison and Duncan test

The paired comparison test known as paired t-test is performed by the T statistic. The least significant difference test (LSD), Duncan test, Tukey test, Dunt test, and Newman Keuls test were used for paired comparisons. In this research, Duncan test was used to determine the conditions of the test and the reasonableness of the data.

A method widely used to compare all pairs of averages is the Duncan multi-domain test (1955). To run this test, the average of the treatments is incrementally ordered and the standard error of each mean is determined as $S_{\overline{V}_{i}} = \sqrt{MSE/n}$. For samples with

different volumes, *n* is replaced by the mean consonant {*ni*}, i.e., $n_h = \frac{a}{\sum_{i=1}^{a} \binom{1}{n_i}}$, then,

with the definition $R_p = r_a(p, f)S_{\overline{y}_i}$, p = 2, 3, ..., a, the comparison is made between the averages. The values $r_a(p, f)$ at the significance level α and f degrees of freedom (equal to the degree of freedom of error) are obtained from the Duncan significant range table.

The test of difference observed between the mean values starts with the difference between the largest and the smallest mean which is compared with the minimum significant range of R_a . Then the test of difference observed between the largest and the second smallest mean is calculated and compared with R_{a-1} , this procedure continues until all the $\frac{a(a-1)}{2}$ average pairs are examined. If *a* observed difference is larger than the minimum relevant domain, the result is that the pair of problem averages have a significant difference.

3 Results and discussion

Surface and deep porosity are structural features of concrete. The presence of porosity in concrete is due to several factors and can be beneficial or harmful. The appearance of the pores on the surface and depth of the concrete can also be either a primary specification or due to secondary interactions (Schmidt et al., 1997). In this study, using comparative images of the topography of concrete surfaces by scanning electron microscope in different conditions before and after testing and measuring the surface porosity according to the proposed method in the previous section, the effect of the variables affecting the corrosion is more accurately analysed and interpreted. Figure 6 shows, for example, the increasing trend of this value over time in a biological pilot.



Figure 6 Comparison of the rate and variation of HIR in a biological pilot (see online version for colours)

3.1 Analysis of laboratory tests

Considering what was said about the reactivity of cement concrete against the acidic environment and the resistance of sulphur concrete, after conducting experiments in a chemical pilot, changes in the HIR in sulphuric concrete seem to be negligible (Figure 7).

Figure 7 Duncan comparison test results in analysing the hole index changes in the chemical pilot (see online version for colours)



On the other hand, according to Figure 8, the surface porosity increase trend due to the dissolution of cementitious polymer and the buckling of concrete cement in two different acidity is clearly visible. The important point the higher slope of the surface porosity in concrete cement at pH of 1, which itself confirms the reason for the further absorption of water by cement concrete, reducing the compressive strength, reducing weight faster and changing the distribution of elements in the surface of the concrete.



Figure 8 Evaluation of the trend of changes in HIR (see online version for colours)





Considering the measurements and data extracted from the biological pilot, it can be said that the microbial environment is at the surface of three-dimensional concrete. The

concrete is porous and is often covered with a porous layer of corrosion. Compounds and micro-organisms are distributed to a level that expands below the corresponding level. The sulphide is dissolved in water and penetrates into the concrete in both phases of the weather. Microbial oxidation is a reservoir for sulphide in total penetration depth. The biological sulphuric acid penetrates into the concrete, and while penetrating into the lower depths, it can neutralise alkalinity. The new concrete has a low permeability and a small portion of its pores is enough to penetrate micro-organisms. However, small interconnecting holes allow the penetration of the dissolved compounds and new bacteria to enter the underlying layers. In this situation, steep chemical surfaces in concrete are rapidly generated. Over time, the porosity of the concrete increases. The dissolution of calcium hydroxide by acid causes the cavities to grow larger and the corrosion process is associated with increased penetration of micro-organisms into concrete. Bock et al. (1988) in Sand (1997) found that concrete up to 6 cm in depth in the Hamburg sewer system was almost destroyed due to bacterial activity. They also discovered that nitrate organisms penetrated the first centimetre of the surface of concrete (Sand, 1997). Figure 9 shows this mechanism in cement concrete.

According to Figure 10 sulphur concrete will also become porous due to elemental sulphur metabolism. However, the porosity rate and pore size ratio are far less than cement concrete. In addition, as shown in this figure, there is an incremental continuous increase in this parameter, measured in both concrete types and in a biological pilot.

Figure 10 Pattern of surface porosity changes over time in each of the concrete (see online version for colours)



The porosity variations in each of the two tested concrete and in both reactors have increased over time and its amount in the biological pilot is higher due to bacterial penetration (Table 1). The significant point about the decreasing trend of porosity in cement concrete in the chemical pilot, in conditions of increasing pH, is the formation of smaller diameter holes (contrary to low pH conditions) therefore, sometimes due to the lack of significant penetration of the sample, a surface layer (though at a low depth) gradually separates from the sample (causing corrosion and surface porosity).



Table 1Evaluation of the changes of HIR in each of the concrete in the chemical and
biological pilot (see online version for colours)

In order to provide more accurate reasoning regarding the occurrence of corrosion in the biological reactor and pH changes in it, modelling the variation of this parameter was described in Figure 11. As shown in the corresponding model, this trend is only affected by time and is defined as a cubic function. The process of pH reduction by the production of biogenic acid and acid neutralisation by dissolving cement compounds can be seen in this model (Vincke et al., 2002).





Secondary pH = $+2.66667 - 0.046168 * \text{Time} + 4.9515\text{E} - 004 * \text{Time}^2$ -1.50892E - 006 * Time³

In order to see the changes caused by chemical reactions and bacterial activities, imaging of the sample surface before and after the experiments at different time intervals presents the interpretations objectively. Figure 12 shows the images of the scanning electron microscope associated with chemical pilot samples with (pH = 1). Magnification of the image of the surface of conventional concrete of 200 micro-metres and 500 micrometres for sulphur concrete was selected. As can be seen, over time, the dissolution of cement compounds and the dissolution of their compounds led to large holes that, the mechanism for its creation was explicitly referred in the preceding sections, while no changes in sulphur concrete in this pilot over time have been observed, obvious porosity is not observed.

Figure 12 Images obtained from a scanning electron microscope for chemical pilot specimens with (pH = 1) (see online version for colours)



Figure 13 The images obtained from the scanning electron microscope associated with chemical pilot specimens with (pH = 2) (see online version for colours)



Figure 14 Images obtained from a scanning electron microscope associated with biological pilot specimens (see online version for colours)



Figure 13 shows the images of the scanning electron microscope associated with the chemical pilot specimens (pH = 2). The main difference between these images and the images of the changes in the concrete cement at pH = 1, the magnitude and type of holes created in this concrete and, as previously mentioned, the uniformity of the porosity at higher pH values is clearly visible.

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. Given the images taken and the time elapsed (Figure 14), the presence of biological strands confirms the presence of bacteria in the concrete surface, especially among microscopic holes. The main difference between these images and the images produced by the chemical pilot are the nature and type of holes formed in a sectional, not widespread in sample concrete surface. Another interesting point is the presence of relatively large depth holes around bacterial strands which is the result of the production of biogenic acid by the gram-negative bacteria of *Thaibacillus thiooxidance* (Harrison, 1984). In the images of sulphur concrete, sulphur decomposition and its metabolism (which is in the hexagonal form) and the appearance of surface asymmetric cracks at the surface of concrete is the main cause of corrosion.

3.2 Analysis of the results of experiments conducted at the sewage treatment plant site

In analysing the images of this section, in addition to using the generality of previous analysis, some principles regarding the microbial physiology of sewage in real and field conditions are mentioned.

220 M.R. Sabour and G.A. Dezvareh

The purpose of this analysis is to prove that the chemical and biological corrosion conditions are different in the actual duct of the sewage with controlled experimental conditions and similarly, different results are expected. There are various limiting or accelerating factors in the corrosion of the two types of concrete that a part of these cases is mentioned. Figure 15 shows bacterial competition in different sewage conditions.



Figure 15 Sequence and coexistence of bacteria present in the sewage and effective in concrete corrosion

As can be seen, under different conditions and long periods of time in the actual sewage system, the possibility of growth and activity of T. *thiooxidans* bacteria as the most effective bacteria affecting the corrosion of concrete specimens is provided when sewage has a long-term stabilised condition (within a few years) and becomes acidic. In such a situation, the corrosion behaviour will be similar to that in the simulated conditions and considering the most severe corrosive state. However, in actual conditions, the presence of flow and alkalinity of input sewage and quality fluctuations and input nutrients makes it almost impossible to make these conditions.

Figure 16 examines the images of concrete samples placed at the entrance to the Shahid Mahallati sewage treatment plant for one year and two months (14 months) from the time of placement. According to the above and the analysis of images, although the corrosion pattern for conventional concrete is almost under the influence of chemical and biological reactions of the sewage, is similar to the laboratory conditions, and especially the biological pilot but the variations in sulphur concrete were completely different and the specimens undergone minimal change that, this important proves the superiority of the use of this concrete in the sewage transfer network.

Figure 16 Images obtained from an electron microscope for specimens placed in a sewage treatment plant (see online version for colours)



222 M.R. Sabour and G.A. Dezvareh

Figure 17 Simulation and schematic interpretation of corrosion in typical concrete specimens (see online version for colours)



Figure 18 Simulation and schematic interpretation of corrosion in sulphur concrete samples (see online version for colours)



As seen in these images, the nature of the bacterial effect in each of the concrete is different and in the same way, the mechanism of decomposition and corrosion is different. In the surface layers of cement concrete, which are adjacent to the biofilm of the bacteria decomposed due to the production of biogenic acid and gradual dissolution occurs, resulting in uniform surface porosity and deep aggregate found in concrete samples. The shape of porosity and holes in cement concrete is circular and with more or less uniform depth. However, according to Figure 19 the bacteria in the sulphur concrete surface affect corrosion by using elemental sulphur for its more favourable metabolism. Hutchinson et al. (1969) found that elemental sulphur caused Thiobacillus thiooxidans (the bacteria used in this research) to grow at a lower pH than the other sub-layer. Many of these species can oxidise thiosulphate to tetrationate. The tetrationate is used to produce pentathionate and tritionate or sulphate, which produces biogenic acid. During these reactions, acid produced, unlike the prevailing conditions in cement concrete, does not affect sulphur concrete. But by sulphur decomposition, cracks are created on the surface that, there is a possibility of sample vulnerability from the cracks. The microscopic images have been clearly shown in Figure 18.

4 Conclusions

After conducting the tests and analyses on the environmental factors of sewage on the corrosion of sewage pipes made of sulphur 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 sulphuric acid condensed in sewage gases or biogenic acid derived from microbial activity by sulphur oxidising bacteria. The process is that by dissolving the cementic polymer and transforming it into etringite and gypsum, the corrosion of concrete samples and pipe crown occur. In this regard, depending on the acidity of the environment, weight loss factors, reduced compressive strength, water absorption, changes in concrete surface elements and surface HIR have been changed and we can use the data from each experiment and develop a numerical model in order to predict the behaviour of the trait in the long period. While in very similar laboratory and field conditions, sulphur concrete exhibited very high resistance to chemical corrosion. The only minor factor of the minor weakness in it and in biological pilot is the elemental sulphur metabolism in the surface layer of the concrete and the depth penetration of the bacteria and the crack occurrence due to the breakdown of the sulphur bond with aggregate material. In these conditions, there were no significant changes in different factors of weight loss, compressive strength reduction, water absorption, surface concrete element changes and surface HIR and progressed at lower rate over time. It should be noted that under the real conditions and the presence of microbial competition between bacteria and the unfavourable conditions for the Thiobacillus thiooxidans bacteria, this malignant effect is far less and is negligible. The results of SEM images also objectively confirm these results.

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