Evaluation of sulfate ions in degrading armed concrete structures of a sewage treatment station: case study

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Abstract
Concrete is susceptible to attack in places considered aggressive, such as wastewater treatment stations. Sulfate ions attack is very common in these places, resulting in accelerated degradation of the concrete, which can compromise any reinforced concrete structure. In this context, the present research aims to evaluate the pathological manifestations caused by these ions, analyzing the sulfate content in concrete samples taken from a station and their porosity. The result obtained in the sulfate content analysis was an average of 7.84%, values well above the acceptable maximum limit that would be on average 0.5%. In relation to the porosity of the sample, an increase of 10% was observed, on average, considering a sample of reinforced concrete without attack.

1 Introduction

The reinforced concrete is the most commonly used construction material in sanitation works [20]. There is increasing concern about the durability of concrete due to the pathological manifestations presented, which compromise the functionality and generate high costs with corrective actions.

Despite studies and technologies developed, water and sewage treatment systems are becoming increasingly aggressive and may jeopardize the durability and operation of sanitation structures [20].

Attack levels vary depending on the environment in which the structure is inserted. The rate of etching varies according to the pH of the aggressive fluid and the permeability of the concrete [15].

According to Mehta and Monteiro [15], sulfate ions are found in natural and industrial environments. Sulfates are ionic chemical species that can cause considerable pathological manifestations in reinforced concrete. Among these manifestations are the expansion and fissure of the concrete, which makes the material more permeable and increases the deterioration process, and consequently, the decrease of the resistance.

In this context, this article aims to evaluate the attack of sulfate ions in a reinforced concrete structure of a Sewage Treatment Station (ETS), analyzing the concentration of sulfate ions in the samples obtained at the site and the porosity of these samples.

2 Theoretical foundation

The water is the fundamental element of attack on concrete structures. Water is generally present in all types of deterioration, and the ease with which it fills the porous solids determines the rate of deterioration [15]. The concrete reinforcement has been the focus of several studies in recent years, including in real structures, not only in the laboratory [2, 16, 27].

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The permeability of concrete, defined by Neville [17] as the ease with which it becomes saturated with water, is the main determinant of its vulnerability to external agents, since the penetration of ions into solution may adversely affect its durability. The permeability of the concrete is not a simple function of porosity, measured as the proportion of the total volume of concrete occupied by pores, but also of pore size, distribution and continuity [17].

Among the various salts that have a deleterious effect on concrete is sulfate, which can be found in natural waters, soil, sea, acid rain, marsh waters, industrial effluents, sewage, among others, and is considered one of the most damaging Agents of a concrete structure [3].
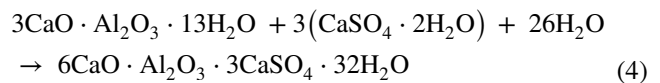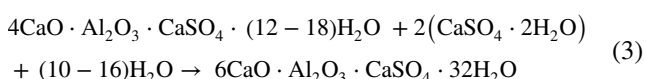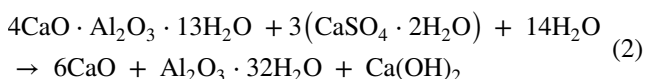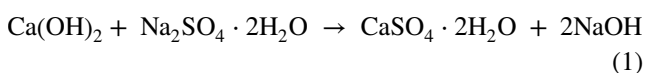
The sulfate may also have internal origin within the structure of reinforced concrete. The sulfate source may be in the clinker due to the various forms of addition of calcium sulfate. The compounds K_2SO_4 and $(K, Na)_3Na(SO_4)_2$ are the most common sulfate phases present in the clinker [24].

In the case of sulfates, there are two known means of attacking the concrete by sulfates: (1) reaction with the hydration products of the unhydrated alumina and/or tricalcium aluminate, producing ettringite; and (2) reaction with the calcium hydroxide producing gypsum. According to Mehta and Monteiro [15], the concentration, the source of the sulfate ions in the water and the composition of the hardened cement paste are determining factors for the predominance of one of the processes.

The intensity of the sulphate attack varies with the cation ion which is bonded to the SO_4^{2-} radical. Although the solubility of sodium sulfates (Na_2SO_4) and magnesium ($MgSO_4$) are higher, the increasing order of aggressiveness begins with calcium sulphate ($CaSO4$), passing through the sulfates mentioned above [3].

2.1 Attack by sodium sulphate (Na₂SO₄)

The attack on the concrete made by sodium sulfate causes two main reactions, which are the formation of gypsum through the reaction of sodium sulfate with calcium hydroxide, as indicated in Eq. 1, and the reaction of gypsum formed with calcium aluminate Hydrated calcium sulfoaluminate or aluminate remaining from the anhydrous cement (C_3A), which form the ettringite, indicated in Eqs. 2, 3 and 4 respectively [25].

$$Ca(OH)_2 + Na_2SO_4 \cdot 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2NaOH \tag{1}$$

$$4CaO \cdot Al_2O_3 \cdot 13H_2O + 3(CaSO_4 \cdot 2H_2O) + 14H_2O$$
$$\rightarrow 6CaO + Al_2O_3 \cdot 32H_2O + Ca(OH)_2 \tag{2}$$

$$4CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot (12-18)H_2O + 2(CaSO_4 \cdot 2H_2O)$$
$$+ (10-16)H_2O \rightarrow 6CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 32H_2O \tag{3}$$

$$3CaO \cdot Al_2O_3 \cdot 13H_2O + 3(CaSO_4 \cdot 2H_2O) + 26H_2O$$
$$\rightarrow 6CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \tag{4}$$

The formed ettringite, after the hardened concrete, occupies a larger volume than the oxide that gave origin, suffering an expansion and consequent cracking of the concrete.

2.2 Attack by magnesium sulphate (MgSO₄)

The fact that magnesium sulphate is more soluble than that of sodium and that of calcium makes it more aggressive to concrete. The attack by magnesium sulphate occurs at an increasing rate. In the reaction there is formation of gypsum and brucite, as shown in Eq. 5. The decalcification of C–S–H due to contact with magnesium sulphate also forms gypsum and brucite [25].

$$Ca(OH)_2 + MgSO_4 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + Mg(OH)_2 \tag{5}$$
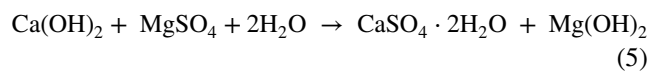
As with sodium sulfate, the gypsum formed reacts with cement hydration compounds to form ettringite. The brucite ($Mg(OH)_2$) and the hydrated silicates from C–S–H decalcification cause the main effect of this attack, which is the formation of M–S–H, hydrated magnesium silicate, a product with low mechanical resistance, indicated in Eq. 6 [25].
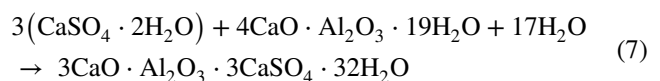
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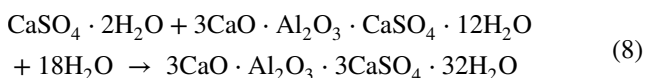$$4Mg(OH)_2 + Si_2O_3 \cdot yH_2O \rightarrow M_4SH_{8,5} + (4,5-y)H_2O \tag{6}$$
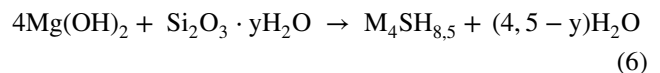
The attack by magnesium sulfate is less than the attack by sodium sulfate, because the brucite layer hinders the diffusion of the solution. However, because C–S–H decalcification occurs, this attack may be more severe, with reduction of the resistance and disintegration of the hydrated cement paste [25].

2.3 Attack by calcium sulphate (CaSO₄)

Calcium sulphate attack is less aggressive than attacks by sodium and magnesium sulphates due to their low solubility in water [3]. Calcium sulphate reacts with hydrated calcium aluminate and monosulfoaluminate, as indicated in Eqs. 7 and 8, to form ettringite.

$$3(CaSO_4 \cdot 2H_2O) + 4CaO \cdot Al_2O_3 \cdot 19H_2O + 17H_2O$$
$$\rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \tag{7}$$

$$CaSO_4 \cdot 2H_2O + 3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O + 18H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \tag{8}$$

In the same way that the attack by other types of sulfate ions, in the attack by calcium sulfate there is formation of ettringite in the already hardened concrete, that generates expansions and consequent fissuration of the structure.

2.4 Sulfate attack on concrete with thaumasite formation

According to Pinheiro-Alves et al. [19], the formation of gypsum and ettringite as the only form of sulphate attack has long been considered. The structure of ettringite and thaumasite are very similar, and are often even confused.

The sulphate attack with formation of thaumasite differs from the attacks mentioned above, with formation of gypsum and ettringite, because it is not the calcium aluminates that are attacked, but the hydrated calcium silicates (CSH), which is the main binding agent of Portland Cement. The hydrated calcium silicates (CSH) are responsible for the cohesion of hardened concrete, including those resistant to sulphates, and contributes directly to the concrete's resistance [4]. As the formation of thaumasite depends on the amount of C–S–H and not on C_3A present in concrete, sulfate-resistant Portland cements (CP-RS), which are low C_3A cements, do not inhibit the formation of thaumasite.

2.4.1 Attack mechanism in an ETE

In concrete in contact with sewage occurs the process of carbonation and acidification of H_2S. The acidification process can reduce the pH of the concrete from 12 to 9, which provides favorable conditions for the propagation of sulfur-based compounds and sulfide oxidizing microorganisms on the concrete surface. These oxidizing microorganisms favor the production of more sulfuric acid, which leads to the formation of two important corrosion products, ettringite and gypsite, according to Eqs. 9, 10, 11 and 12 [6].
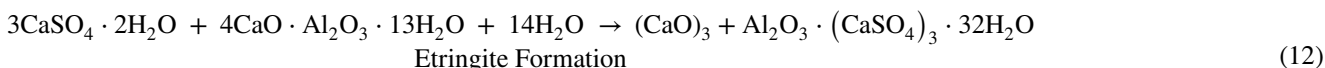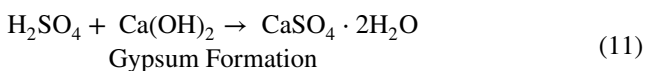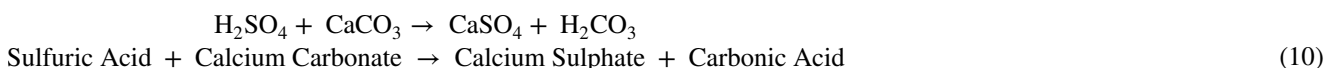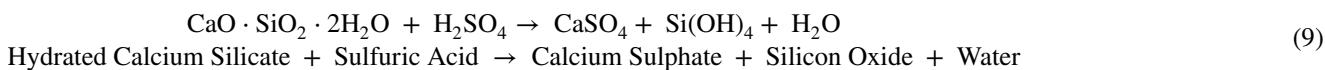
Gypsum and ettringite are expansive compounds, which can cause cracking and can trigger a series of pathological manifestations.

The study of the effect and neutralization of H_2S was done by Jiang et al. [7] and Joseph et al. [9]. Already Sharma et al. [23] have performed a modeling of the effect of the pH on the transfer of the existing H_2S in the liquid medium to the gaseous medium. The effect of the combined attack of sulfates with other ions has been studied by Zhang et al. [28], Maes and Belie [13] and the study of corrosion in reinforced concrete structures was done by Jiang et al. [7] and Jiang et al. [8], and Hoppe Filho et al. [5] have studied the use of mineral additions as a sulphate mitigating agent.

3 Materials and methods

In order to reach the specific objectives of this work, two laboratory tests were carried out, one to determine the sulfate content in the concrete and another to analyze the porosity of the sample, both of which are described below.

3.1 Specimen collection

The samples tested were collected from the Santa Quitéria Sewage Treatment Station (STS), located in the Barigui River sub-basin, which serves, in whole or in part, the 18 districts of Curitiba, PR, besides the municipalities of Araucária and Campo Magro.

The sewage treatment at this station is carried out through physical, chemical and biological processes, which remove solids, organic matter and nutrients.

The Sewage Treatment Station Santa Quitéria was inaugurated in 1998, and has the capacity to handle an average flow of 520 L/s and can reach 600 L/s. It has an anaerobic treatment process, with six fluidized bed anaerobic reactors, type UASB, shown in Fig. 1, and post treatment system by flotation, which was inaugurated in 2010.

$$CaO \cdot SiO_2 \cdot 2H_2O + H_2SO_4 \rightarrow CaSO_4 + Si(OH)_4 + H_2O$$
$$\text{Hydrated Calcium Silicate} + \text{Sulfuric Acid} \rightarrow \text{Calcium Sulphate} + \text{Silicon Oxide} + \text{Water} \tag{9}$$

$$H_2SO_4 + CaCO_3 \rightarrow CaSO_4 + H_2CO_3$$
$$\text{Sulfuric Acid} + \text{Calcium Carbonate} \rightarrow \text{Calcium Sulphate} + \text{Carbonic Acid} \tag{10}$$

$$H_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4 \cdot 2H_2O$$
$$\text{Gypsum Formation} \tag{11}$$

$$3CaSO_4 \cdot 2H_2O + 4CaO \cdot Al_2O_3 \cdot 13H_2O + 14H_2O \rightarrow (CaO)_3 + Al_2O_3 \cdot (CaSO_4)_3 \cdot 32H_2O$$
$$\text{Etringite Formation} \tag{12}$$

The Sewage Treatment Station also has gas scrubbers to control bad odors. For a while, the reactor tanks were capped, as shown in Fig. 1, in an attempt to minimize the bad odors, but since the concrete was rapidly deteriorating, they were uncovered.

During the visit to this Sewage Treatment Plant, four samples were taken, one from each tank, of the deteriorated part of the concrete of a UASB reactor, which at the time of the visit was deactivated for routine cleaning. Figure 2 shows the layout of the Sewage Treatment Station with indication of the Sampling place, as well as in Fig. 3, the sampling tanks.

The structural elements of the tanks that make up the reactor were visibly damaged, as illustrated in Fig. 4, so it was possible to manually withdraw the samples.

Figure 4a shows an overview of the interior of one of the sample collection tanks, while Fig. 4b shows the deterioration of the concrete due to the sulphate attack, and Fig. 4c shows the sample removed from the site, that due to high porosity and low resistance of the concrete, it was possible to make the collection manually.

With a visual analysis it is possible to observe the deterioration of the concrete and the corrosion of the reinforcement, as indicated in Fig. 5.

It should be noted that Rheinheimer and Khoe [22] extracted testimony from the structure and verified that the compressive strength of the concrete used is 42.2 MPa. According to the sanitation company specification, the cement used is CP-IV, according to current Brazilian standards, which has up to 35% of fly ash added and the water/cement ratio is at most 0.45.

The collected samples were duly packed and sent to the Laboratory of Cement Chemistry of the Federal Technological University of Paraná, Campus Curitiba, where the tests were carried out.

Fig. 2 Layout of Sewage Treatment Station (Source: Google Earth. Accessed on: 12/05/16)

3.2 Test of sulfate content

The chemical test to determine the sulfate ions content in the concrete samples was carried out according to an adaptation of the standard "4500-SO$_4$—Sulfate" of the Standard Methods for the Examination of Water and Wastewater [26] for waste water. This procedure recommends to submit the ground sample, whose diameter is less than 850 µm, to a solution of hydrochloric acid with barium chloride, which will form a precipitate of barium sulphate, which after calcination at 800 °C is heavy.

To determine the percentage of sulfur trioxide in the sample, Eq. 13 was used,

$$SO_3(\%) = \frac{34.3 \times M_{calcined}}{M_{Standard}} \tag{13}$$

where: $M_{calcined}$: Mass of the weighed sample after the test in grams; $M_{Standard}$: Mass of the Standard sample before the

Fig. 1 UASB type reactors (Source: http://site.sanepar.com.br/ Accessed on: 12/5/16)

Fig. 3 The sampling tanks (Source: The author (2016))

Fig. 4 Collection of samples (Source: The author (2016))
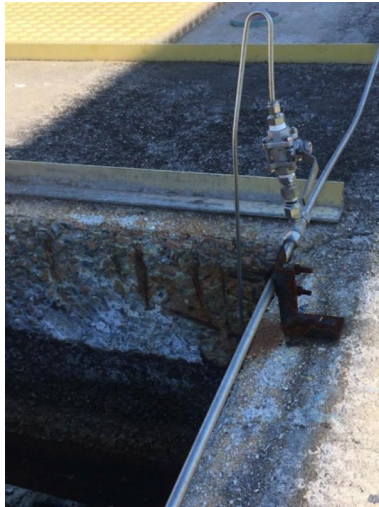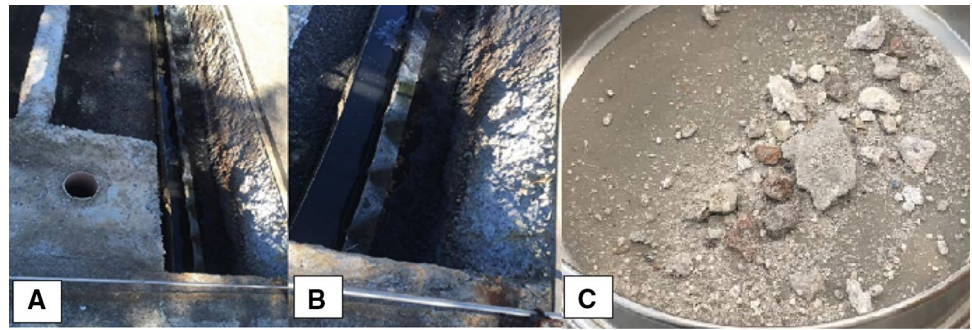
Fig. 5 Reinforcement corrosion (Source: The author (2016))

Fig. 6 Quantachrome Poremaster 33 (Source: http://www.quantachrome.com/ Accessed on: 12/05/16)

test, in grams; $SO_3(\%)$: Percentage of sulfur trioxide in the sample.

3.3 Analysis of sample porosity

To analyze the porosity of the sample, the Mercury Injection Porosimetry method was used. The technique is based on the fact that mercury behaves like a non-wetting fluid in relation to most substances, so was not spontaneously penetrate small holes or cracks in these materials unless pressure is applied to it. [10].

The equipment used was the Poremaster 33, from Quantachrome, which can be visualized in Fig. 6, and whose technical data are in Table 1.

The assay was performed on a 0.17 g sample. The procedure consists of placing the sample in a container and applying a low pressure to the liquid, which allows the filling of most of the large pores. As the pressure increases, the small pores are also filled. With the values of the pore volume of the concrete penetrated by the mercury and with the respective pressure applied, a porosimetric curve is obtained.
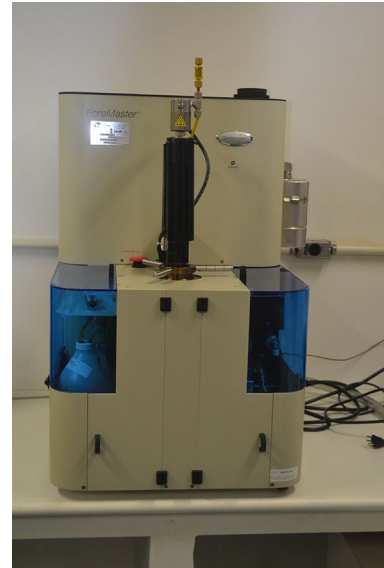
4 Results and discussions

The test for determining the sulfate content in the concrete sample carried out in this work resulted in the formation of a white precipitate, barium sulphate, and the sulfur trioxide content was determined. The results obtained with this assay are shown in Table 2.

In Table 2 it can be observed that the result of sample 2 probably presents some test error, thus it was disregarded in the analyzes. The valid results show an average of 7.84% and a standard deviation of 1.04% as sulfate content in the tanks of the UASB reactors of this Sewage Treatment Station. Considering that the test works with a small amount of sample, a confidence interval for the mean was established, assuming a normal distribution for the results, and the interval of 6.08% was calculated as the minimum value and 9.60% as the maximum value of the sample confidence interval for the mean.

To determine the actual amount of sulphates that penetrated the structure, a sample of an uninjured region was

Table 1 Technical data of Poremaster. Source: Quantachrome Instruments [21]

The equipment—technical data	
Manufacturer	Quantachrome
Model	Poremaster 33
Gas	Synthetic air at 60 psi
Environmental temperature	20 °C
Equipment pressure reading	
Minimum pressure	0.2 to 50 psi
Maximum pressure	20 to 33,000 psi
Maximum cell diameter	
d	10 mm
h	20 mm
Mercury DATA	
Contact angle	140°
Superficial tension	480 dynes/cm

Table 2 Result of the sulphate determination test. Source: The author (2016)

	Mass$_{standard}$ (g)	Mass$_{calcined}$ (g)	SO$_3$ (%)
Sample 1	1.115	0.252	7.75
Sample 2	1.110	0.589	18.20
Sample 3	1.144	0.298	8.93
Sample 4	1.107	0.221	6.85

drawn into the outside of the UASB reactor tank. For this case, the value of 0.58% was calculated, and as observed in Table 2, all the samples tested had higher values, that is, the contamination of the concrete by sulfate ions occurred. The values found are close to those obtained by Lorente et al. [12] in the laboratory, which ranged from 2.8 to 6.2% for sodium and magnesium sulphates, respectively, in a concrete with 50.2 MPa. Already Mazer et al. [14] evaluated the pillars of a bridge where they plotted the sulfate profile, whose values were 1.6% to 0.3% for depths of 5 mm and 15 mm. Liu et al. [11], analyzing the tunnel concrete, observed sulphate concentrations of 12.1% and 16.5%.

In this Article was desired to analyze the amount of sulfate present in the concrete, and not in the sewage that the structure maintains direct contact. However, Rheinheimer and Khoe [22] carried out a sulfate concentration quantification test on the sewage sample of this same Sewage Treatment Station, which was collected at the same site, and the result was less than 200 mg/L of SO$_4^{2-}$, With adjusted dilution.

According to NBR 12655 [1], an amount of sulfate present in water of less than 200 mg/L indicates a weak or moderate exposure condition due to aggressiveness. For the European standard, this amount of sulfate falls in the class I of environmental aggressiveness. For both standards, this value means that the risk of deterioration of the structure is insignificant or small.

Although the concentration of sulfate ions present in the water indicate a small aggressiveness, was observed that the Sewage Treatment Station in study presents a high degree of deterioration of its concrete structures and a high amount of sulfate present in the concrete sample, which are indicative that the aggressiveness Is high, for this reason it is believed that TEE should be considered as chemically aggressive industrial environments whose risk of deterioration of the structure is high, that is, class IV of environmental aggressiveness.

Sanepar, however, classifies the reactors as environmental class IV, whose aggressiveness is very strong, and the risk of deterioration is high. This classification is consistent with reality. Still, other specifications should be made, such as the use of sulfate-resistant cements.

Considering that the sulfate ion attack increases the porosity of the concrete structure, the porosity of the sample of concrete attacked by sulfate ions was compared with a sample that was not attacked. The results of the Mercury Injection Porosimetry test showed that total porosity of the sample of concrete attacked by sulfate ions is approximately 12.76%, while a sample of non-attacked concrete with a compressive strength of 40 MPa, resistance required by Sanepar for the construction of Sewage Treatment Station, presented a porosity of 2.45%, this is similar a porosity observed by [15]. With all that has been analyzed it is possible to affirm that there was a considerable increase in the porosity of the concrete.

During the porosimetry test, for complete filling of the pores with mercury, was necessary to increase considerably the pressure applied in the liquid. The relationship between pressure applied on the mercury and the diameter of the pores can be seen in Fig. 7. The dashed line consists of the division between low and high pressure, and this value corresponds to 31.074 PSI.

Based on Fig. 7, was observed that 43.75% of the pores were filled with mercury at a low pressure, and thus, to fill the other 56.25% needed to apply high pressure, which reached 31,304.36 PSI. With this, it is concluded that most of the pores are very small.

Another analysis was the relationship between the pore diameters and the intruded volume of mercury, which can be observed in Fig. 8. Thus, the higher the volume of intruded mercury, the smaller the pore diameter.

The Analyzes of Figs. 7 and 8, was possible to conclude that the pores with larger diameters are first filled with a low pressure, and for full filling of the pores, a considerable pressure increase was required. Consequently, the higher the volume of mercury intruded in the sample, the smaller the pore diameters.

Considering the high porosity measured in the samples, the high sulphate concentrations observed are indicated, as

Fig. 7 Pressure applied to mercury versus pore diameter (Source: The author (2016))
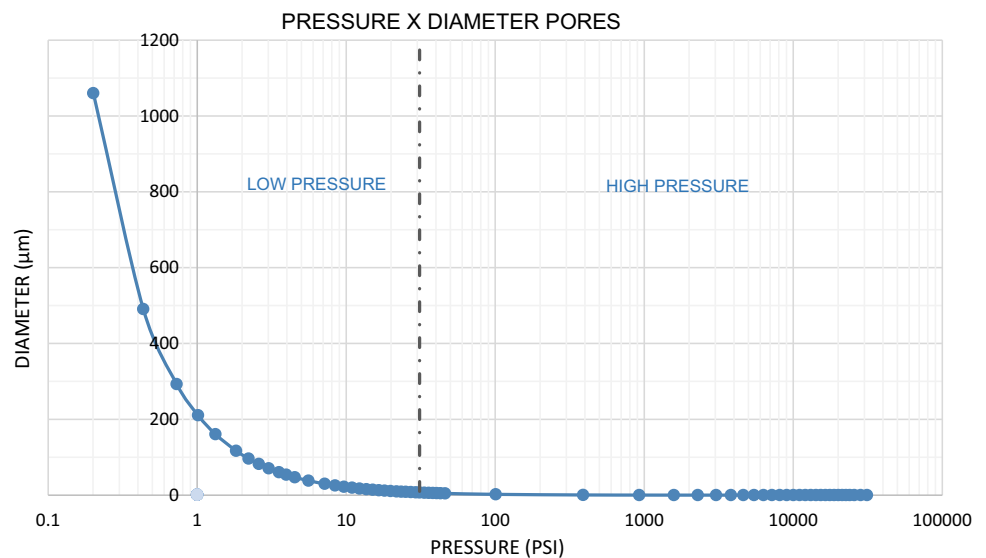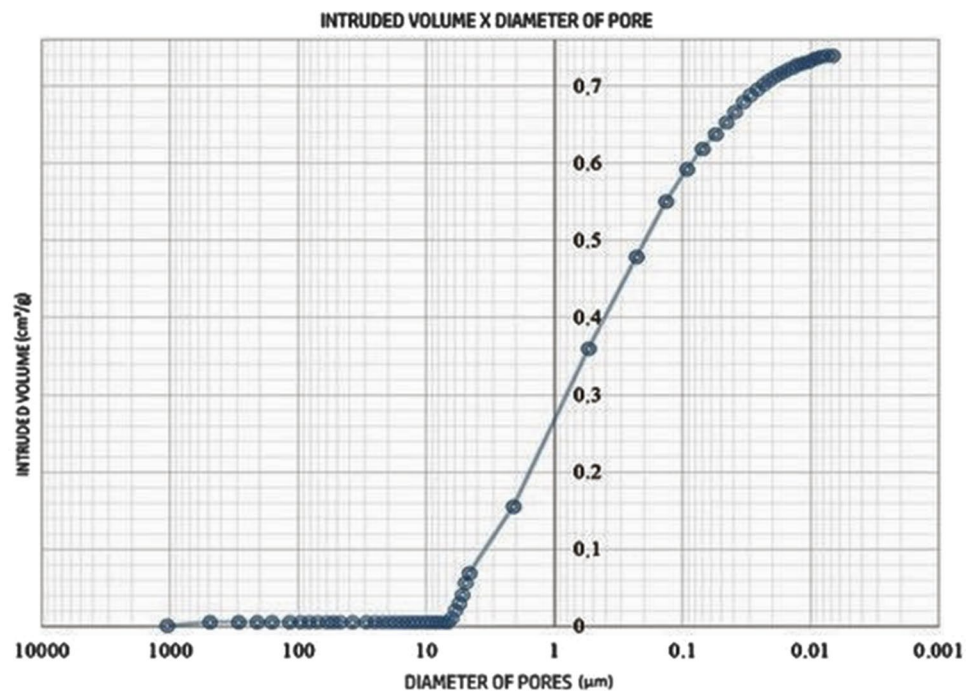

Fig. 8 Intruded volume versus pore diameter (Source: The author (2016))

indicated in Table 2, as well as the deterioration found at the site, shown in Figs. 4b, 5, where surface deterioration and corrosion of the reinforcement are shown.

5 Conclusions

This article aimed to evaluate the pathological manifestations caused specifically by sulfate ions in a sewage treatment plant, was observed that the reinforced concrete structure was in an advanced state of deterioration.

The content of sulfate ions found in the concrete samples tested indicates contamination of the concrete of the TEE, even with levels considered normal of sulfate ions in the waste water stored in the tank.

One of the damages caused to the concrete by the attack on the sulfate ions is the increase in porosity, which could be observed with the mercury injection porosimetry test, where the contaminated sample had a porosity approximately 6 times greater than an uncontaminated sample and with the same compressive strength.

The results indicate that the values of the sulfate concentration in the sewage are in accordance with the literature, and present a high risk of deterioration of the concrete of the Sewage Treatment Stations.

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