

Evaluation of Corrosion Caused by Biogenic Sulfuric Acid (BSA) on the Concrete Structures of Sewerage Systems (Chemical Tests)

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Abstract—The research studies of the kinetics of the corrosion process that attacks concrete and occurs within sewerage systems agree on the amount of variables that interfere in the process. This study aims to check the impact of the pH levels of the corrosive environment and the concrete surface, the concentrations of chemical sulfuric acid, and in turn, measure the resistance of concrete to this attack under controlled laboratory conditions; it also aims to contribute to the development of further research related to the topic, in order to compare the impact of biogenic sulfuric acid and chemical sulfuric acid involvement on concrete structures, especially in scenarios such as sewerage systems.

Keywords—Acid Sulfuric, concrete, corrosion.

I. INTRODUCTION

CONSIDERING the diverse environmental conditions to which concrete structures in sewerage systems are exposed, it is important to know the causes, consequences and solutions that can apply to corrosion caused by the action of biogenic sulfuric acid (BSA); for this it is necessary to note that the first evidence or record of corrosion in sewage systems was documented by Olmstead and Hamlin in 1900 [1]. This phenomenon is intense and has a great economic impact, in countries such as Germany 40% of 100 billion dollars are spent in repairing damages caused by BSA; in Flanders (Belgium), the biogenic corrosion represents a cost of 10% of the total cost of wastewater treatment. In Los Angeles (USA), about €400 million is spent on the restoration of pipelines affected by BSA [2].

Concrete is a construction material that looks like rock; it is a material composed of a coarse granular material, embedded in a cement paste, which serves as glue for the materials [3], and, in turn, is one of the most used materials in sewerage systems because of its high strength, low cost, great durability compared to other materials, and at the same time holds greater volumes of water compared with PVC and metal tubes. Evidence has shown that corrosion is present in many concrete structures related used with water and in wastewater treatment.

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The alarming fact is that some of these facilities are significantly deteriorating after less than a decade of use [4].

The corrosion process that occurs in concrete structures by the action of sulfuric acid, mainly in the pipes of sewerage systems, water treatment plants and inspection chambers, is known by different names such as: biogenic sulfuric acid corrosion, hydrogen sulfide corrosion and microbial induced/influenced corrosion [5].

There are many countries in the world which generate sulfuric acid, as shown in Fig. 1 it is applied in different aspects and uses such as: phosphate fertilizers, mineral processing, petroleum alkylation, pulp and paper, synthetic plastics, chemicals, nylon processing, detergents, metal sulfates, batteries, boiler water treatment, ethyl alcohol and more. It is also used as a raw material and input in industrial processes due to its acidifying power.

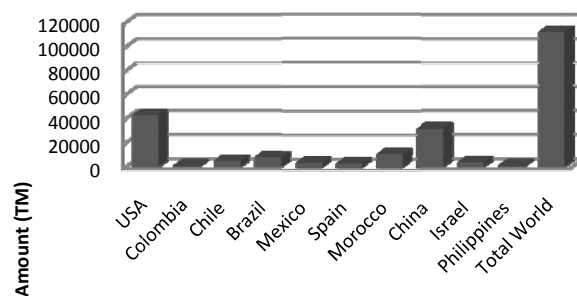


Fig. 1 World Production of Sulfuric Acid, Thousands of TM

II. METHODOLOGY

This research project is based on a quantitative methodology, since it responds to hypothetical deductive parameters, and seeks to analyze by experimental means, under controlled conditions, the phenomenon of corrosion that occurs in sewerage systems. To conduct the research, the theoretical and practical procedures are established to determine results.

According to the above, the proposed experiments indicate that the concrete specimens are immersed in three sulfuric acid solutions with different concentrations; the weight of each concrete specimen is taken before immersion on an electronic balance accurate to $\pm 0.10\text{kg}$, as well as the moisture levels and compressive strength of the three specimens. The solutions are prepared with distilled water in three cylindrical tanks ($\phi = 1,03\text{m}$, $h = 0.73\text{m}$) with a capacity of 500L, the

concentrations of SA at 98% are 1%, 5% and 10%. 257.9 L of solution is prepared in each tank as shown in Table I, as well as the amounts of solute and solvent, with the greatest amount of SA being used in the 10% solution with a total of 232.11 Liters (L) of this chemical component.

TABLE I
AMOUNTS OF SOLUTE AND SOLVENT USED TO PREPARE THE SOLUTIONS

Solution Concentration	1%	5%	10%
Volume of Solvent in Liters (L)	255.321	245.005	232.11
Volume of Solute (H ₂ SO ₄ Al 98%) in Liters (L)	2.579	12.895	25.79

The pH and temperature of the solution are recorded before immersing the concrete specimens. The duration of the test period is two months; during this period the pH and temperature values of the solution with the specimens are measured, three times a day, to establish the changes that occur. The data collection is performed with a pH meter (brand HANNA - instruments reference HI 8013); a piece of equipment that measures temperature, conductivity and pH of solutions with a reliability level of 95%.

In each tank, the concrete specimens are placed, on a plastic base, in a grid pattern. The base is placed in order to prevent accumulation of particles, which are produced by the acid attack on the concrete surface, around the bottom of the cylinders. Subsequently, the proposed exposure time is waited before performing the tests and evaluating the corrosion of concrete cause by this phenomenon.

III. EXPERIMENTAL DESIGN

Sulfuric acid is an aggressive chemical agent that has the ability to change the properties of strength and durability of concrete structures in sewerage systems. In order to propose the series of experiments the number of experiments, it is necessary, as a first step, to make a list of variables and classify them as dependent and independent, as can be seen in Table II. In this way, it is easier to determine which variables can be controlled by the people conducting the tests.

Chemical corrosion tests on concrete specimens have a reliability rate of 95%, in which the techniques and events will be applied several times expecting the same result in all the phases; likewise, to ensure a high level of reliability in the data it will be necessary to have homogeneous conditions when applying the tests or measurements, in other words, at the same time, in the same place, with the same analysts and with the same equipment.

Regarding the validity of the data, the criteria used is the predictive model because the obtained data will have to be correlated with each other, and also the calculated variance should not mark a high value and should not generate a significant error in the respective analysis.

TABLE II
CHEMICAL TEST VARIABLES

Variable	Dependent	Independent
Environment Generation Chemical Tests		
Ph Solution	X	
Temperature		X
Sulfuric Acid Concentration (Chemical Tests)		X
Physical Characteristics of the Specimens		
Exposure Time		X
Dimensions of the Cylinder		X
Resistance of the Cylinders		X
Ph of the Concrete Surface		X
Mix Type (Standard Mixture for Pipes and Mix for Marine Environments)		X
Cylinder Weight		X
Type of Reinforcement (Graphite, Metal Fibre, Polymer Fibre)		X

Should not mark a high value and should not generate a significant error in the respective analysis.

For this reason, a certain number of specimens are used to be exposed to the environment, where the two variables (exposure time and concentration) are the most important factors for calculating a representative sample used for the tests.

The concrete cylinders shall be exposed to the following types of tests:

- Compressive strength.
- Mass loss.
- Permeability.
- Electrochemical tests (corrosion in the metal reinforcements).
- Detection of corrosion in the concrete.

The concrete specimens are 48 per tank, because two types of mixtures (24 cylinders of a standard mixture for pipes and 24 cylinders of a mixture resistant to sulfates for marine environments) will be used. For each of the two compositions, 5 samples will be used for the compression test, as a representative sample is needed that is reliable for a subsequent calculation of averages, to obtain a low random error, so for three exposure times 15 cylinders are needed for this test. To calculate the permeability after exposure to the environment, 3 cylinders supported in NTC Standard 4483[6], are needed, and as 3 times are specified 9 cylinders are necessary to have reliable data in this test; and therefore 54 concrete cylinders are needed for exposure with the 3 concentrations per mixture, and this is the experimental arrangement of the two levels with mixtures without reinforcement.

Initial analysis of the specimens will be performed with eight cylinders, 5 of which will be for the compression test, which also serve to measure the initial mass of the specimens; and 3 for the permeability test, with the results being used to compare the impact of chemical sulfuric acid at 98% in concrete specimens according to the type of mixture.

The amount of concrete cylinders to be used during the chemical tests are 160 of $\varnothing = 10\text{cm}$ and $h = 20\text{cm}$, which must

be handled appropriately to avoid affecting the reliability of the data.

Solutions of 1, 5 and 10% will be implemented in short tanks of 500 L. To calculate the amount of solute and solvent it is necessary to predict the volume displaced by the 48 cylinders upon being introduced into the tanks volume, and the solution volume is 257.9 L and the volume of the solute varies in each of the tanks.

The factorial arrangement in this experiment phase consists of all the possible combinations of the levels and the treatment factors, in the tests of mass loss and strength it is a 3x3x2x5 arrangement; and in the permeability test the arrangement is 3x3x2x3. These combinations are useful to generate the number of specimens that should be used for a specific phase of experiments.

IV. MATERIALS

A. Concrete Test Specimens

In the tests, cylindrical concrete specimens are used, which are used to assess the corrosion in the concrete mixtures; they have a diameter of 10 cm and height of 20 cm. All the concrete specimens are prepared under the NTC 550 [7], standard and will be supplied by the cement manufacturing company S.A. TITAN, with the specifications of the mixture used for making concrete pipe and a mixture for marine environments that is resistant to sulfate attack; these mixtures are dry with a low water cement ratio, to obtain a concrete with high density, low absorption, low permeability and long durability, produced under NTC Standard 1022 [8]. The cylinders used have a drying period of 28 days and a resistance of 350 Kg/cm². A total of 160 cylinders of $\phi = 10\text{cm}$ and $h = 20\text{cm}$ are exposed to chemical attack.

B. Sulfuric Acid

The sulfuric acid that is used has a purity of 98%, has a brownish colour, is a highly corrosive mineral acid and is an oily liquid. It is available in aqueous solutions of between 33 and 98%, its properties can be seen in Table III; the acquisition of this chemical in Colombia involves various permits and certificates required by the State in order to handle this substance. All this is carried out through the Ministry of Justice, as it is the body in charge of and responsible for ensuring that this tool is used for a proper purpose.

TABLE III
 PHYSICAL PROPERTIES OF SULFURIC ACID AT 98%

Property	Value
Molecular Weight (G/Mol)	98.08
Physical State	Liquid
Boiling Point (°C, 760 Mmhg)	310-335
Melting Point (°C)	3
Specific Density At 15.6°C	1,844

There are two methods of industrial manufacture or production of sulfuric acid; the lead chamber process and the contact process. This acid is used as a catalyst in the

manufacture of fertilizers, herbicides, glue, oil purification, in the leather industry and as an agent in the production of explosives.

C. Water

The water that is used to prepare the solutions is distilled, because a high level of purity so that the acid does not react with other minerals contained in drinking water from the aqueduct of the municipality of Tunja. The distillation process guarantees the removal of minerals, and if done correctly, the water should contain only oxygen and hydrogen molecules, and have a pH level of 7.

V. SAFETY EQUIPMENT

The handling of sulfuric acid is a process that requires individuals to protect themselves from this chemical, which is why safety equipment is a mandatory requirement so that no part of the body is affected by this substance, and is necessary in order to conduct this research. The minimum protective equipment necessary can be seen in Fig. 2 and it includes:

- Plastic masks which function to protect the face, which is the body part most susceptible to damage during the handling of SA.
- A complete set of rubber clothing (trousers and jacket) is responsible for safeguarding the skin; rubber gloves are also used for the hands for a better handling of this substance.
- Rubber boots resistant to chemicals, which are certified against any risk and also include a steel toecap to protect the toes from blows

The storage of sulfuric acid is carried out in a suitable cabinet for this purpose, with the key only being accessible to people who will conduct the project and who have the proper knowledge of the dangers of this product. Signs are placed all around the laboratory, but in places near to the sulfuric acid it is increased indicating the substance and the risk that a person faces if they are not sufficiently cautious. Notices are placed on the access door leading to the tanks, where the tests are carried out in the mentioned project, and the information placed there is preventive and informative.

As a precautionary measure, a fire extinguisher should be placed at the exit to the room where the experiments are carried out, and should have an adequate distribution in order to minimize risks. The labelling of the substance includes information about the associated risks and actions to be taken in an emergency; according to current international legislation for the handling of chemicals, a notice is placed at the entrance to the testing room with all these specifications of the ICSC reference: 0362 IPS (International Programme on Chemical Safety): 0362 of the IPS (International Programme on Chemical Safety).



Fig. 2 Personal Protective Equipment

VI. TESTS TO BE PERFORMED

A. Compressive Strength Test

The compression test of the concrete cylinders is conducted under NTC Standard 673[9]. Prior to testing, the cylinders are subjected to a process of cleaning, brushing and drying. The capping that is used is neoprene in accordance with NTC Standard 504[10]. The equipment used for the test is a digital hydraulic press with a reliability level of 95.45% [11]-[12].

B. Permeability Test

The method for the permeability test is specified in NTC Standard 4483[13]; this test aims to determine the permeability coefficient K , by applying a pressure to one face of the specimen equivalent to 0.5 MPa, for 4 days. Once the specimen is saturated, and the flow has been adjusted to the opposite side, successive measurements of the volume flow are made to verify that the flow has been constant.

Once the flow is established to be constant, the volume of water passing through the specimen in a given time period is determined. Once the volume flow has been determined, the permeability coefficient of the concrete is calculated using (1):

$$K = \frac{\rho L g Q}{P A} \quad (1)$$

where:

K : Coefficient permeability in m/s.

ρ : Density of water in kg/m³.

L : Length of the specimen in m.

g : Gravity in m/s²

Q : Water flow rate in m³/s.

P : Water pressure in N/m².

A : Cross sectional area of the specimen in m².

C. Test to Determine the pH of a Concrete Surface

NTC Standard 3689[14], describes the method for determining the pH of a concrete surface attacked by an acid. The process described by the Standard is performed with litmus paper. However, there are other methods to record the pH variations in materials; one of those is the phenolphthalein test.

Phenolphthalein is a colorimetric pH indicator. It is an organic molecule which has two different structures depending on the pH, one of which is stable at an alkaline pH above 9, and the other at an alkaline pH less than 9. The test uses a 1% phenolphthalein solution in alcohol and is applied by spray or drops on the concrete surface; if the surface becomes pink, the pH is less than 9.

VII. DATA COLLECTION AND ANALYSIS

The indicated time for the collection of data depends on when the specimens are introduced into the tanks, given that one hour too long or one hour too little in the exposure of the cylinders can affect the reliability of the data.

All data will be organised in Excel due to its wide applications in areas such as data analysis, probability and statistics, and an ANOVA analysis will also be performed to determine the variance of the data and its reliability level.

The ANOVA is a very flexible method that allows statistical models to be created for the analysis of experimental data whose values have been observed in various circumstances. Essentially, it is a method for dividing the variance of the dependent variable in two or more components, each of which can be attributed to an identifiable source (variable or factor); in turn, the well-known analysis of variance is the most accurate method for calculating the variability of a measurement system, because it has the advantage of quantifying the variation due to the interaction between the operators and the parts.

VIII. CONCLUSION

- The corrosion of concrete structures is very common in the world; it is a silent phenomenon that must be fought because of the large amount of money that is lost by performing ongoing maintenance works.
- The experimental design in a research project based on laboratory tests is essential because it identifies the number of treatments that are done during the practice phase.
- The number of cylinders per tank in each of the solutions of 1, 5 and 10% are 48, and in total for the experimental phase are 160 specimens; this amount is substantial and reliable for the respective tests.
- When handling sulfuric acid, it is necessary to equip oneself with safety equipment such as masks, overalls and boots due to the danger of the chemical agent.
- The reliability of the data depends on the calibration of the instruments used in the laboratory tests, and it equally depends on a constant period of time for each of the respective tests.

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