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# DEGRADATION OF ORDINARY PORTLAND CEMENT BASED CONCRETE IN SULPHURIC ACID

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#### Abstract

It is now well acknowledged that, countries with hot climates are not the only places where concrete sewer pipes are susceptible to corrosion from sulfuric acid attack. The seriousness of this problem is only understood when the pipe bursts, causing surface flooding and other negative effects. There aren't many published laboratory models and experimental studies that look at the fundamental factors that control corrosion, though. Therefore, the purpose of this study was to determine the degradation of concrete built using Ordinary Portland Cement (OPC) with varying water to cement ratio in sulphuric acid were affected by the exposure conditions (flowing and static) and acid concentration (pH). In all conditions, concrete samples were cast and exposed to sulfuric acid at pH levels of 1 and 2. Compressive strength and mass loss were used to gauge concrete deterioration. After the testing time, it was discovered that the mass loss and compressive strength loss were greater for the flow of sulphuric acid, the gypsum generated becomes dissolved, making crystallization harder and limiting growth. Moreover, deterioration accelerated when the pH dropped from 2 to 1.

*Keywords*: Sulphuric acid attack; Ordinary portland cement; Mass loss; Compressive strength; pH..

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## Introduction

Concrete is the most essential building material that the average person is familiar with. It has been used for centuries to create infrastructure such as buildings, roads and highways, wastewater systems, and much more. When concrete is subjected to acid attack, its durability is called into doubt. Sewage transportation and collection are typically done through concrete structures, making them more prone to deterioration [1-2]. This deterioration is multistage and complicated in character. Olmstead and Hamlin initially proposed this idea in 1900 [3], when they observed erosion of cement lining in an outfall sewer after just 5 years of installation. They investigated the entire process and came to the conclusion that the corrosion was mostly caused by sulphuric acid, which led in the creation of gypsum, which created fissures. C.D. Parker [4] also investigated sewage corrosion and linked it to bacterial activity inside the sewer network. Corrosion of concrete in sewer is caused when the sulphates in the sewage are reduced by sulphate reducing bacteria (SRB) under anaerobic condition. The SRB helps in the formation of hydrogen sulphide  $(H_2S)$  gas. As  $H_2S$  is lighter than air it evaporates to aerobic atmosphere where aerobic bacteria are present [5]. These bacteria oxidized the H2S into sulphuric acid (H<sub>2</sub>SO<sub>4</sub>). This process is known as biogenic corrosion of concrete sewers [6]. The corrosion of concrete in the sewer caused by the generation of sulphuric acid decreases the life expectancy of the sewage structure from 20 years to 15 years and in extreme cases to 10 years [7]. Because of the difficulty of access, structures corroded by acids are always ignored as it is associated with high costs and unneeded interruptions. As a result, when corrosion damage is discovered, the structure is already in disrepair and necessitates extensive repairs.

Although there is ample information about sulfuric acid's attack on concrete, the main focus of these works is either the water-tocement ratio or the type of cement. Also, the investigations were either conducted in a static condition or employing the brushing approach to speed up the procedure. experimented conducted Various by researchers [7-9] etc. have used brushing technique in their respective research works to calculate degradation of concrete. While [10-12] etc. has worked in static condition. Very few scientists have used the flowing condition in their studies [13-15] are among them; they tested the disintegration of concrete by putting it under flowing circumstances in sulphuric acid. There haven't been any previous research known to author that quantitatively demonstrate the distinction between a flowing situation and a static one at various pH levels. Cement concrete cubes manufactured with OPC were therefore cast and exposed to pH 1 and 2 under various environmental circumstances in order to achieve the purpose of this investigation.

## MATERIALS AND METHODS

## Raw materials

All the material used in this study was locally procured. Ordinary Portland Cement (OPC) was the principal binding agent used. The fine aggregate was composed of local river sand. Well-graded crushed stone aggregates with a maximum aggregate size of 20 mm were used as coarse aggregate. A poly carboxylic superplasticizer with a specific gravity of 1.04 and a weight proportion of 0.5 percent was added to cement to improve its overall workability.

## Mix proportion and sample preparation

According to the guidelines outlined in IS 10262: 2009 [16], the mix design was completed. Table 1 describes the mix proportions in detail. An electrically driven concrete drum-style mixer was used to combine the ingredients. As the initial step in the mixing process, all of the aggregates were first added to the mixer. To make sure the aggregates were evenly distributed, the components were then dry mixed for 30 seconds. After adding only half of the water, the mixture was mixed for a further two minutes. The mixer was then turned off for a bit to allow the aggregates to soak up the water. After that cement was added and the mixer was run for 30 seconds and the remaining 50% of the water was added. Concrete cubes of 150x150x150 mm<sup>3</sup> were created in appropriately lubricated cast-iron moulds. The mould was filled with three layers of concrete before any trapped air

was released by vibrating the mould for two minutes on an electrical table. The cast samples were kept in the lab for 24 hours without being touched. After 24 hours, concrete samples were demoulded, and they were stored in a curing tank with fresh water for an additional 28 days. The water in the tank was replaced every seven days.

Table 1 Quantities of Mix Design						
Designation	Water	Coarse	Fine Aggregate	Plasticizer		
	cement ratio	Aggregate (kg/m <sup>3</sup> )	(kg/m <sup>3</sup> )	(% by weight of cement)		
M1	0.38	1100	675	0.5		
M2	0.40	1100	675	0.5		
M3	0.42	1100	675	0.5		

#### **TESTING PROCEDURE**

#### **Exposure** condition

The concrete samples were put inside four containers containing sulphuric acid solutions. Figure 1 illustrates the experimental configuration. Two of the tanks were made for the flowing action of sulphuric acid solution, whilst the other two tanks were made under static conditions. The static exposure tanks were not touched during the entire procedure. A motor with a pumping system powered the tanks that were intended to be in a flowing state. For the eight hours it ran every day, the motor gave the sense of flowing. A detailed experimental schedule can be seen in Table 2.



Fig. 1 Experimental setup

The chemical reaction between the solution in the tanks and the concrete during the exposure procedure caused the pH to alter. A pH metre was used to monitor daily pH variations, and acid was supplied when necessary to maintain the concentration at the desired level. The specifics of the experimental strategy are detailed in Table 2.

Table 2 Details of Experimental Plan					
Desig	nation	Surrounding Condition	Mix	рН	
M1 pH 1 F	M1 pH 2 F	Flowing	M1	1,2	
M1 pH 1 S	M1 pH 2 S	Static		1,2	
M2 pH 1 F	M2 pH 2 F	Flowing	M2	1,2	
M2 pH 1 S	M2 pH 2 S	Static		1,2	
M3 pH 1 F	M3 pH 2 F	Flowing	M3	1,2	
M3 pH 1 S	M3 pH 2 S	Static		1,2	

## Mass Loss

Samples were taken out of the tanks after a period of 1, 3,5,7,9, and 12 months of immersion and pat dried before being put in an oven for 24 hours for 105°C until consistent mass was reached. The dry mass  $(M_t)$  of the samples subjected to various exposure conditions of sulphuric acid solutions was recorded using weighing balance having an accuracy of 0.01g. This is to mention here that the samples were also weighted in oven dry condition. The percentage mass loss was then calculated as follows:

Mass Loss % =  $(M_0-M_t)/M_0 \times 100$ 

Where,

M<sub>0</sub>= Dry mass of sample before subjecting to acid attack

M<sub>t</sub>= Dry mass of sample at particular time after subjecting to acid attack

## Compressive Strength

The compressive strength of all the concrete samples was evaluated by testing in an automatic compression testing machine in accordance with IS 516:1959 [17]. The samples were tested under load control mode at a loading rate of 400 kg/min. For all the ages, three replicate samples were tested for each exposure condition and average values recorded as

the compressive strength. Prior to testing, the samples were taken out of the tank and pat dried before being immersed in water for 48 hours. The samples were tested immediately after being removed from water.

## **Results & Discussions**

## Mass Loss

The mass loss of concrete samples with different water to cement ratio subjected to flowing and static exposure conditions at various pH levels is compared in Figure 2. When compared to static conditions, the rate of increase in mass loss was higher for flowing conditions. This might be explained by the solution's shearing action. The flow of an acidic solution produces shearing motion. The acidic solution's shearing action removes all of the hydration products that have built up on the concrete's surface. The hydration products mainly gypsum which is developed as the reaction between solution of sulphuric acid and the silicates present in the cement. Every time the acidic solution runs, the most built-up material on the surface is eliminated, creating a fresh surface for the attack [13]. The same findings have been made in an experiment by Kawai [12], Kong [14], and Fytianos [15]. The study's findings concur with those of the cited writers. As opposed to the samples in the flowing condition, the sample in the static condition lost less mass. This is caused by the accumulation of

reaction

hydration

hydration products in the structure's pores and microscopic fissures [18]. Surface scaling develops over time as a result of



these

expanding

products [12, 19].

Fig. 2 Mass loss of concrete in different surroundings at various pH (a) 1 (b) 2

Also, from the figure 2 it can be seen that the mass loss in concrete specimens made with M1 and M2 began immediately after immersion, whereas mass loss in concrete specimens made with M3 increased over the first 30 days. This is most likely due to sulphuric acid absorption. Several researchers relate the better resistance of concrete to sulfuric acid attack to raising the water cement ratio, which is lower in concrete with a higher water cement ratio than in concrete with a lower water cement ratio [20]. It was also said that such an improvement may be attributable to the fact that the volume percentage of cement for low water cement ratio concrete is greater than that for high water cement ratio concrete. Another theory is that sulfuric acid primarily affects calcium hydroxide, which is said to be more plentiful in cement pastes with low water cement ratios.

Furthermore, regardless of the environment, as shown in figure 2, pH 1 has a higher amount of deterioration. This is due to the fact that acidity affects concrete strength, as demonstrated by Kawai and Fytianos [12,15].Therefore, it can be concluded that the rate of concrete deterioration caused by sulfuric acid is significantly influenced by the sulfuric acid solution's concentration..

#### Compressive Strength

The compressive strength of concrete samples with different water to cement ratio exposed to flowing and static exposure circumstances with pH 1 and 2 is compared in Figure 3.Figure 3 demonstrates that for both solutions, the strength loss differential during flowing exposure conditions (i.e., pH 1 and pH 2) is greater than under static conditions. The development of hydration products is to blame for this disparity. Hydration products accumulate on the specimen when it is in a static state. As a result, these materials enter the concrete structure's pores under the pressure of accumulation. The hydration products that have collected on the surface and in the pores of the concrete specimen provide some resistance against breaking in order to provide the specimen with some modest structural support [21].



Fig.3 Compressive strength of concrete in different surroundings at various pH (a) 1 (b) 2

Also, as seen in the figure 4 the drop in compressive strength is more for high water to cement ratio as compared to low water cement ratio. This is attributed to the fact that in case of high water to cement ratio a network of capillary pores forms and is well connected throughout [22]. There is greater seepage detected because the water is conveyed via these knit network when it flows. While in cases of low water to cement ratio, hydration products restrict the capillary pores. The general effective media hypothesis (GEM) states that when there is a high water to cement ratio, water passes via both capillary and gel pores. The interaction between capillary pores and gel pores determines how permeable such materials are which ultimately lead to loss of strength.

Here also, as shown in figure 3, pH 1 has a higher amount of deterioration. Again this is due to the concentration that affects concrete strength, as demonstrated by Kawai and Fytianos [12,15].

## Conclusions

1. Concrete submitted to the flowing state lost more mass and compressive strength than concrete submitted to the static condition, indicating that the creation of hydration products acts as a barrier, slowing down the deterioration process.

- 2. The compressive strength test should not be used to determine the durability of concrete in an acidic environment since production of hydration product mainly gypsum increase the compressive strength was seen under static circumstances as compared to flowing one.
- 3. Acid concentration plays an important role in deciding the degradation of the concrete. As the acidity increases more degradation was observed.

## References

- 1. Sand W., Importance of hydrogen sulfide, thiosulfate, and methylmercaptan for growth of Thiobacilli during simulation of concrete corrosion, Applied Environmental Microbiology, June 1987, Vol.53, No. 7, pp. 1645– 1648.
- 2. Sand W. and Bock E., Concrete corrosion in the Hamburg sewer system, Environmental Technology Letters, December 1984, Vol. 5, pp. 517–528.
- 3. Olmstead W. and Hamlin H., Converting portions of the Los Angeles outfall sewers into a septic

tank, Engineering News, 1900, Volume 44, pp. 317–318.

- 4. Parker C., The corrosion of the concrete I. The isolation of a species of bacterium associated with the corrosion of concrete exposed to atmospheres containing hydrogen sulfide, Australian Journal of Experimental Biology and Medical Science, 1945, Volume 23, pp. 81–90.
- 5. De Belie N. Monteny J. Beeldens A. Vincke E. Van Gemert D. and Verstraete. W., Experimental research and prediction of the effect of chemical and biogenic sulfuric acid on different types of commercially produced concrete sewer pipes, Cement and Concrete Research, 2004, Volume 34, pp. 2223-2236.
- Roberts D.J. Nica D. Zuo G. and DavisJ.L., Quantifying microbially induced deterioration of concrete: initial studies, International Biodeterioration & Biodegradation, 2002, Volume 49, pp. 227–234.
- 7. De Muynck W. De Belie N. and Verstraete W., Effectiveness of admixtures, surface treatments and antimicrobial compounds against biogenic sulfuric acid corrosion of concrete, Cement and Concrete Research, March 2009. Volume 31, No 3.
- Vincke E. Verstichel S. Monteny J. and Verstraete, W., A new test procedure for biogenic sulfuric acid corrosion of concrete, Biodegradation, 1999,Volume 10, No. 6, pp. 421-428
- Yang Y. Ji T. Lin X. Chen C. and Yang Z., Biogenic sulfuric acid corrosion resistance of new artificial reef concrete, Construction and Building Materials, January 2018,, Volume 158, pp. 33-41
- 10. Ma. Guadalupe D. Gutiérrez-Padilla. Bielefeldt A. Ovtchinnikov

S. Hernandez M. and Silverstein J., Biogenic sulfuric acid attack on different types of commercially produced concrete sewer pipes. Cement and Concrete Research, February 2010, Volume 40, No 2, ,pp. 293-301

- 11. Grandclerca A. Danglab P. Gueguen-Minerbea M. and Chaussadenta T., Modelling of the sulfuric acid attack on different types of cementitious materials, Cement and Concrete Research, 2010, Volume 55, No 33 pp. 126-133
- 12. Kawai K. Yamaji S. and Shinmi T., Concrete Deterioration Caused by Sulfuric Acid Attack'. 10DBMC International Conference on the Durability of Building Materials and Components, LYON, France
- 13. Hudon E., On the biodeterioration of concrete sewer pipes- a practical analytical model, Thesis submitted to, Montreal for PhD, University of McGill 2014.
- 14. Kong L. Lu H. Fu S. and Zhang, G., Effect of corrosion layer on the deterioration of concrete in gravity sewers, Construction and Building Materials, November 2020, Volume 272, pp.121663
- 15. Fytianos G. Tsikrikis A. Anagnostopoulos C.A. Papastergiadis E. and Samaras P.,The Inclusion of Acidic and Stormwater Flows in Concrete Sewer Corrosion Mitigation Studies Water, January 2021, Volume13, No.3
- 16. Indian Standard specification for concrete mix proportioning — Guidelines, IS 10262-2019. Bureau of Indian Standards. New Delhi
- 17. Indian standard methods of tests for strength of concrete.IS 516-1959 (Reaffirmed in 2004. Bureau of Indian Standards. New Delhi
- 18. Mahmoodian M. and Alani, A.M., Effect of temperature and acidity of

sulfuric acid on concrete properties, Journal of Materials in civil Engineering, 2017, Volume 29

- 19. Mehta P.K. and Paulo J. M. M. *Concrete:* Microstructure, *Properties, and Materials*', MC Graw Hill, New York.
- 20. Hewayde,E., Nehdi,M., Allouche,E. and Nakhla,G., Effect of mixture design parameters and wetting-drying cycles on resistance of concrete to sulfuric acid attack, Journal of Materials in Civil

Engineering, March 2007 Vol, No. 19 pp. 155-163.

- 21. Boyd A.J. and Mindess S., The use of tension testing to investigate the effect of W/C ratio and cement type on the resistance of concrete to sulfate attack, *Cement and Concrete Research*, March 2004, Volume 34, No.3 pp. 373–377
- 22. Cui and Cahyadi J.H., Permeability and pore structure of OPC paste, Cement and Concret Research, Vol. 31, No. 2, pp. 277-282