Performance of concrete under aggressive wastewater environment using different binders

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Abstract: The aim of this study was to investigate the influence of cement composition in aggressive wastewater environments. For this purpose different mortar prisms were prepared using CEM-I (Ordinary Portland Cement), CEM-II (Portland Limestone Cement), SRPC (Sulfate Resisting Portland Cement) and GGBS (Ground Granulated Blast-Furnace Slag). The prisms were cured for 28 days and then were immersed in 5% Na2SO4 solution for 4 months. During this period expansion was measured by estimating the change in length after each 28 days. The Laboratory results have shown that the prisms containing GGBS having less expansion than the prisms containing CEM-I or CEM-II. A slight improvement in performance relative to SRPC binder was also observed. The XRD (X-ray Diffraction) analysis was also performed on the mortar prisms to determine the gypsum or ettringite formations. From the XRD graphs, the most notable peaks of Gypsum and Ettringite correspond to prisms manufactured using CEM-I, CEM-II and SRPC. Presence of sulfuric acid in the wastewater is creating the main problems for the concrete structures. To find a suitable solution to this problem, concrete cubes were prepared using different binder ratios of CEM-I, CEM-II, SRPC and GGBS. These cubes then cured for 28 days. Cubes were divided into two groups of six each. Six cubes were placed in water and the other six were dipped into acid solution for about 4 months. The Mass variation and compressive strengths were recorded after every 28 days. Experimental results have indicated that the cubes containing GGBS showed different behavior from the cubes prepared by CEM-I and CEM-II. The compressive strength of the GGBS cubes was higher than the other cubes. The experimental results show that addition of GGBS to concrete structures improves the strength of the structures and enhances their resisting capacity against aggressive wastewater environments.
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1. Introduction

The deterioration of wastewater infrastructure has long been a concern but the issues surrounding the problem remained unknown for many years. Research has tended to focus on the deterioration of concrete in sewer systems and pipelines (Mori et al. 1992, Ayoub et al. 2004, De Belie et al. 2004). There is clearly a need to differentiate between the two forms of attack, sulfate attack and sulfuric acid attack on concrete structures. So a detailed research has been conducted to check the effects of corrosion and deterioration on the concrete structures. Concrete sewers tend to be corroded by the wastewater. So the performance of the concrete sewers is largely dependent on the mix design. Existing research has shown that corrosion is present in many concrete structures associated with water and wastewater treatment. The serious problem about the concrete structures is that some of these structures are deteriorating after less than a decade in service. From the latest research it has been made clear that current design practices may not be appropriate to deal with the aggressive nature of the wastewater. Sulfate attack is one of the most frequent and damaging phenomena that contribute to the deterioration of cementitious materials. External sulfate attack is caused sulfates from ground water, soils, solid industrial waste and fertilizers, from atmospheric SO2, or from liquid industrial wastes. Availability of these sulfates to cause damage to concrete depends on their concentration and solubility, transport of water, and environmental conditions. External sulfate attack on concrete is not yet completely understood (Cohen & Mather 1991, Metha 1992, Brown & Taylor 1998). Some researchers concluded that limestone addition to the concrete mix could increase the sulfate resistance, while other researchers, found a decrease of sulfate resistance depending on the replacement level and clinker composition (Soroka & Stem 1976, Zelic et al 1999).

Durability is an important engineering property of concrete, which determines the service life of concrete structures. The durability of concrete reduces when interaction with acids and alkalis occurs. So it can be accepted as a general rule that acids are deleterious to concrete. Acidic attack usually originates from industrial processes, but it can even be due to urban activities. Extensive experiences have shown that addition of GGBS to cement improves the durability and performance of the concrete structures in severe environment (Bejin 1998). In recent years, more optimized high performance Portland cements blended with pozzolanic materials have been introduced for concrete structures in severe environment. These additions have considerably improved the behavior of the concrete structures in wastewater environments.

Another major problem for wastewater infrastructures is the presence of hydrogen sulfide in collection systems. It also causes some serious problems which are of major concern. Hydrogen sulfide is a nerve gas and is toxic in the concentration ranges that can occur in sewers. To avoid damages and problems due to hydrogen sulfide many safety concerns have been developed. Hydrogen sulfide has a characteristic smell often associated with the smell of rotten eggs. Hydrogen sulfide is not the only odorous gas in sewer networks; however, as hydrogen sulfide is easy to measure, it is also often used as marker for sewer odor, although other compounds also contribute to the odor (Hwang et al, 1995).

The main goal of this investigation is to adequately compare the corrosive phenomenon and to give proper solutions to these problems. This study was conducted to improve the concepts about the processes involved in sulfate resistant cement and GGBS induced concrete corrosion and deterioration. To achieve the main objective, experiments have been conducted to determine the mechanisms and reactions of the concrete surfaces induced with sulfate resistant cement and GGBS.

2. Literature Review

The research on the issue of the deterioration of the concrete structures is being started since from a long time and many research articles have been published. Literature shows that this issue has focused on three distinct topics in the sulfate/sulfuric acid effects on concrete as shown below (O'Connell et al 2010):

i. Studies of the biological processes behind the corrosion of wastewater infrastructure, with particular reference to the role of sulfate-reducing and sulfur oxidizing bacteria.
ii. Studies of the chemical effects of sulfates and sulfuric acid on concrete mixes
iii. Laboratory based research methodologies, especially those incorporating the biological effect on concrete.

Sulfate attack is caused by reactions of numerous cement components with sulfates originating from external or internal sources. The presence of magnesium sulfates in soil and ground water can reduce the service life of concrete structures. In a solid state these sulfates are harmless to concrete; however, once present in solution above a certain threshold they can have serious effects on concrete. These sulfates then produce expansion, cracking, spalling and loss of strength in the concrete structures. The attack occurs when the sulfates present in the water react with calcium aluminates (C3A) present in the cement and phases formed during hydration such as calcium hydroxide. However, the general attack is associated with the formation of gypsum and ettringite, which is reported to result in large expansions (Lea 1998). The use of different binders as cement replacement materials has been well documented with respect to sulfate resistance (Freeman et al, 1995). Materials such as fly ash and GGBS can also improve the transport properties of the hydrated material by reducing permeability, which also increases the sulfate resistance of cement based materials (Lea 1998). The primary effect of ground slag admixtures on the properties of the freshly mixed concrete is to provide better workability. It results lower w/b ratios for the concrete structures (Hogan & Meusel 1981). Concrete mix proportioning for optimum performance with the slag can be accomplished in accordance with ACI Committee recommendations (Raphael 2010). Both laboratory testing and field experience have shown that properly proportioned slag Portland cement concretes have the following properties compared to regular Portland mixes (Lewis 1981):

i. Higher ultimate strengths with a tendency toward lower early strengths
ii. Higher ratio of flexural to compressive strengths
iii. Improved refractory properties
iv. Lower coefficients of variation in strengths
v. Improved resistance to sulfates and seawater
vi. Lowered expansions from alkali silica reactions

Corrosion of concrete caused by hydrogen sulfide has been recognized as a serious problem in collection systems for the past century (Olmsted et al 1990, Parker 1947, Okabe et al 2007). The problem of hydrogen sulfide induced concrete corrosion is well known in known in wastewater collection systems (Zhang et al, 2008). In some of the worst cases, the lifetime of sewer pipes and pumping stations has been reduced to less than ten years (Hvitved-Jacobsen 2002). Standards consider ettringite formation as the only sulfate related risk for the durability of hydrated Portland cement. Expansive ettringite formation in hydraulic concrete (due to sulfate attack) can be prevented, avoiding thereby its destructive effect.

3. Materials and Methods

The concrete mixes were prepared and tested to investigate the relative resistance of the cementations binders to the aggressive wastewater environments. The selected binders and their compositions are listed below in the Table 1. The binder combination selected for different samples are listed in table 2. The following locally available materials were used for this research:

- Sand (Lawrence Sand Pit)
- Cement (CEM I, Ordinary Portland Cement) (CEM II, Limestone Portland cement) (SRPC, Sulfate Resisting Portland Cement)
- GGBS (Ground Granulated Blast-Furnace Slag)
- Coarse Aggregate having size 10 mm and 20 mm.
- Water
- Sulfuric Acid
- Sodium Sulfate.

Table 1: Chemical composition of binders

Chemical Composition (mass%)	Binders Types			
	CEM I	CEM II	GGBS	SRPC
SiO_2	20.8	19.8	35.1	21.3
Al_2O_3	4.8	4.8	12.4	3.5
Fe_2O_3	2.7	3.1	0.6	4.1
CaO	64.3	62.8	40.6	63.3
MgO	0.7	1.9	8.6	2.1
Mn_3O_4	0.1	0.2	0.4	0.1
Na_2O	0.1	0.2	0.3	0.2
K_2O	0.7	0.6	0.4	0.6

The test methods are completed according to the standards. Standard solutions of Sodium sulfate and Sulfuric acid are made and the nature of the attacks of these solutions is examined. Sulfates are present in the sewage but the sulfuric acid is not readily available in the wastewater. The sulfuric acid is the product of the metabolic process of thiobacillus bacteria. The following tests were conducted on the samples:

i. Sodium sulfate expansion tests
ii. Sulfuric acid tests
iii. X-Ray Diffraction Analysis (XRD Test)

Table 2: Binder combinations chosen for testing

Mix designation	Binder composition
MA	100% CEM II
MB	50% CEM II + 50% GGBS
MC	30% CEM II + 70% GGBS
MD	100% CEM I
ME	30% CEM I + 70% GGBS
Na_2O	0.1
K_2O	0.7

Sodium Sulfate Test

A modified ASTM C1012 procedure was used to test the mortar prisms for change of length when exposed to a sulfate solution. Six types of different Mortar Prisms of dimensions 285mm x 25mm x 25mm were prepared through use of the standard mix in EN196-1 for cement conformity testing. Each mix contained 450g of binder, 1350g of sand (Lawrence Sand pit) and 225g of water for the production of four prisms. The freshly cast specimens were placed in a moist air cabinet at 20˚C and de-molded after twenty-four hours. After that they were immersed in a water bath at 20˚C and allowed to cure until an age of twenty-eight days. The standard exposure solution used in this test method contains 50g of sodium sulfate (Na2SO4) per liter of water. Each solution was prepared with 4.5 liter of water and mechanically stirred until fully dissolved. The solution was then topped up with water until a volume of 5 liter was achieved.

The prisms were stored for a period of four months and the solution was refreshed on a monthly basis. Comparator readings were taken every four weeks. The readings consisted of taking an initial reference measurement for each prism and a standard reference measurement prior to immersion in the sodium sulfate solution. The change in length of the prism is recorded with reference to the initial reading and is then calculated using Eq.1:

$$\Delta L = L_X - L_I / Lg \qquad \text{Eq.1}$$

Where;

ΔL = change of length at age 'x' (%)
Lx = comparator reading of specimen at age 'x'
Li = initial comparator reading of specimen
Lg = nominal gauge length or 250mm as applicable

\The percentage change of length of each prism was expressed to an accuracy of 0.001% and the average of the four test specimens was recorded.

Sulfuric Acid Test

In this method six different concrete mixes having 12 cubes each were prepared to expose the environment similar to those found in wastewater treatment plants. Concrete cubes of 150×150×150 mm in size were made with different water binder ratios. The cubes casted were placed in air for twenty four hours. Then all of the cubes were stored in a curing tank at 20˚C for twenty-eight days following which the strength of cubes 1 and 2 for each mix was tested under compression until failure. The remaining ten cubes were now divided into two sets of five split between continued storage in the curing tank and immersion in a 1% sulfuric acid solution for up to 112 days. All cubes were weighed every seven days for the first month and every twenty eight days thereafter to record any mass loss or mass gain. The strength of the remaining ten cubes was also taken after each twenty eight days. The sulfuric acid solution for all experimental schedules was maintained at a pH of approximately 1.5 by titrating the solution with a more concentrated sulfuric acid to keep the pH within a margin of ± 0.3. The solution was refreshed once a month to avoid prolonged contamination associated with the corrosion products of degraded concrete.

3. Experimental Results

Sulfate Resisting Test

Laboratory results of the mortar prisms for all binder combinations were taken for a period of about four months. The results are shown in the figure 1.It is clear that the mixes prepared using only CEM I and CEM II have shown greater expansions. They showed about same trend of expansion regarding the other samples. The mixes prepared using CEM II with 50% and 70% GGBS outperformed the all other samples by showing less expansion. However CEM II + 50% GGBS showed minor discoloration effect but in case of CEM II + 70% GGBS not showed any visual evidences of attack. The sulfate resisting Portland cement was worst in performance after four months. Visually it has begun to exhibit the same common degradation phenomenon when approaching 0.030–0.035% expansion, namely cracking, spalling and a white speckled appearance. So the analysis shows that CEM I binder is not classified as suitable for "severe" or "very severe" exposure classes, While CEM II and SRPC binders are not suitable for "very severe" exposure classes.

It can be concluded that within the given testing parameters, there is a clear benefit to the addition of a high percentage (≥ 50%) of GGBS to mortar. This would seem to be the case regardless of the cement type used although it would appear to have a greater contribution in increasing the resistance of CEM I mixtures to sulfate attack. The CEM II, limestone cement used in this experimental programme exhibits an inherent sulfate resisting capability although may not be sulfate resistant. When combined with a percentage of GGBS greater than or equal to 50%, the resulting mixture exhibits a superior level of sulfate resistance.

Visual Inspection

Expansions were observed for a period of four months for all six mixes. Early exposure up to 84 days for specimens MA, MB, MC, ME and SR showed an identical trend of expansion showing little expansion. However MD was already showing greater expansion in comparison and this mix was showing continual expansion phenomenon. At 112 days MA showed an inferior resistance to the aggressive solution compared with MB, MC, ME and SR. Both mixes MA and MD contain no addition of GGBS. Mix MD is ordinary Portland CEM I cement while mix MA represents Portland CEM II limestone cement. There were some marked differences in visual deterioration of the mixes varying from corrosion related deposits and discoloration to cracking and warping of the specimens, or a combination of each. The severity of the visual deterioration corresponds well to the degree of expansion observed. Examination of each of the test mortar prisms indicated the formation of longitudinal cracks around 0.035-0.04% and this was common to all mixes. The OPC specimens (MD and ME) show a similar crack formation although lacking in any white substance. The remaining specimens containing GGBS (MB and MC) have not yet shown any crack formation. Longitudinal cracking along the length of the specimens was not an exclusive mechanism with radial cracking observed on one of the MA specimens along the boundary of the reference stud. One of the other visual distinctions between MA and MD were notable depositions of a white substance occurring in blotches at random intervals on one of the prisms. Produced cracks in these specimens are shown in figures 2-5. These deposits seemed to be an integral part of the paste and were not soft to touch, nor had they the ability to be removed by scratching the surface.

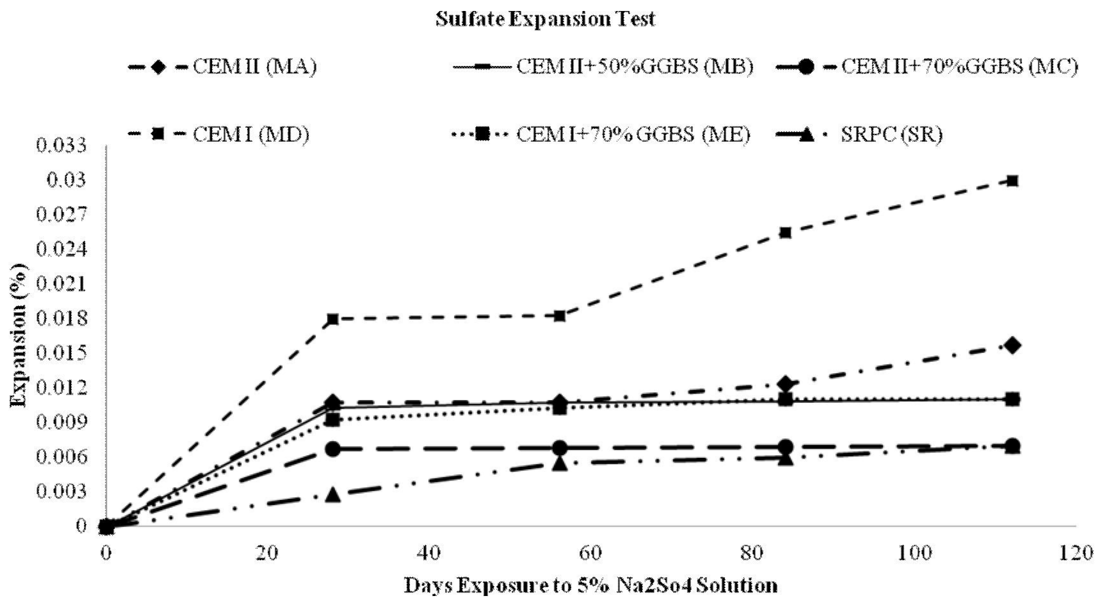


Figure 1: Results of sulfate expansion test

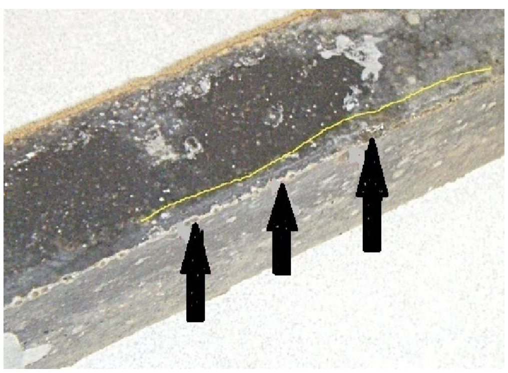


Figure.2: White filled crack in SR Specimen

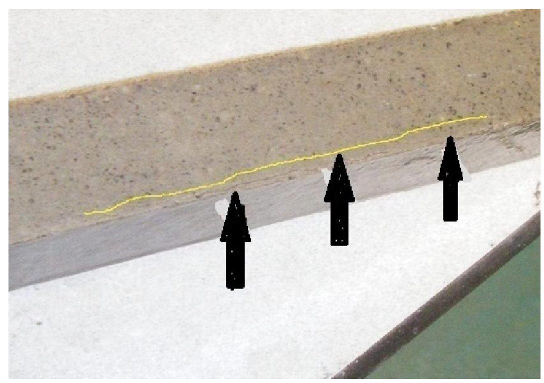


Figure 4: Longitudinal crack in ME specimen

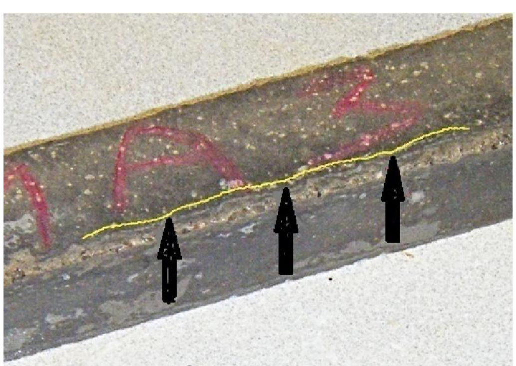


Figure 3: White filled crack in MA Specimen

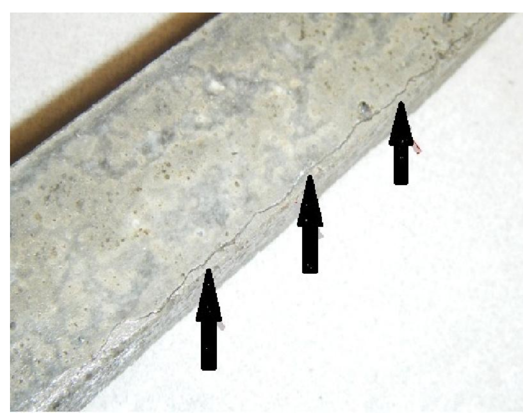
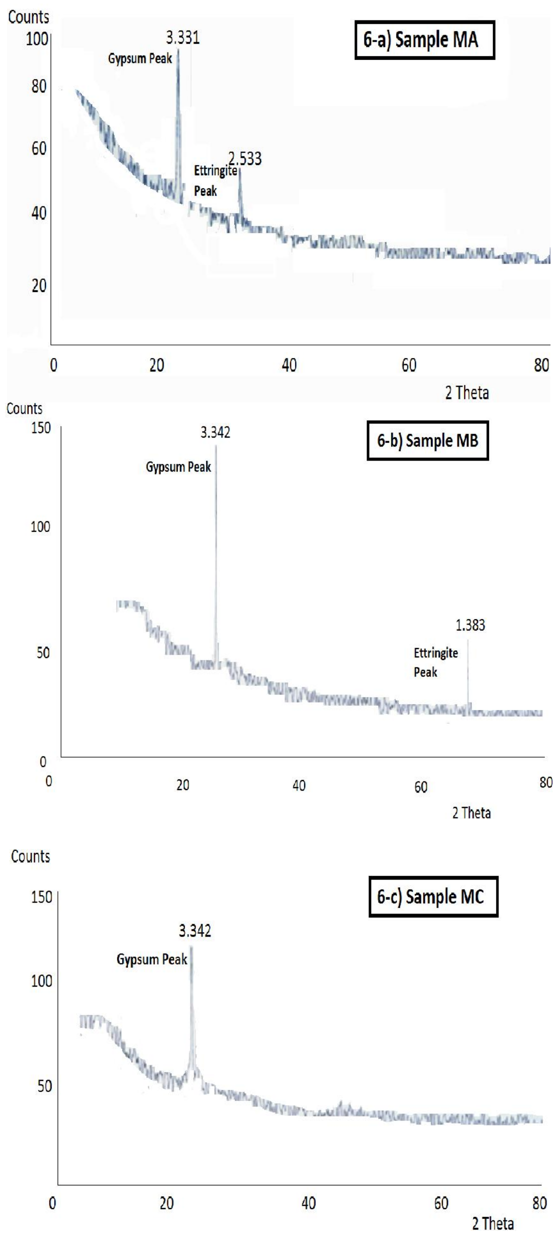


Figure5: Longitudinal crack in ME specimen

XRD Analysis

The main purpose for this test was to examine the behavior of reaction between the various cementitious binders and the sulfate solution. For this purpose samples were taken from those mortar prisms which were used in the sulfate expansion test at the end of the expansion test. The samples were then powdered using grinding or polishing machine. This powder was then examined using X-ray diffractometer. The results have been scanned and shown in figures 6a, 6b, 6c, 6d, 6e & 6f.



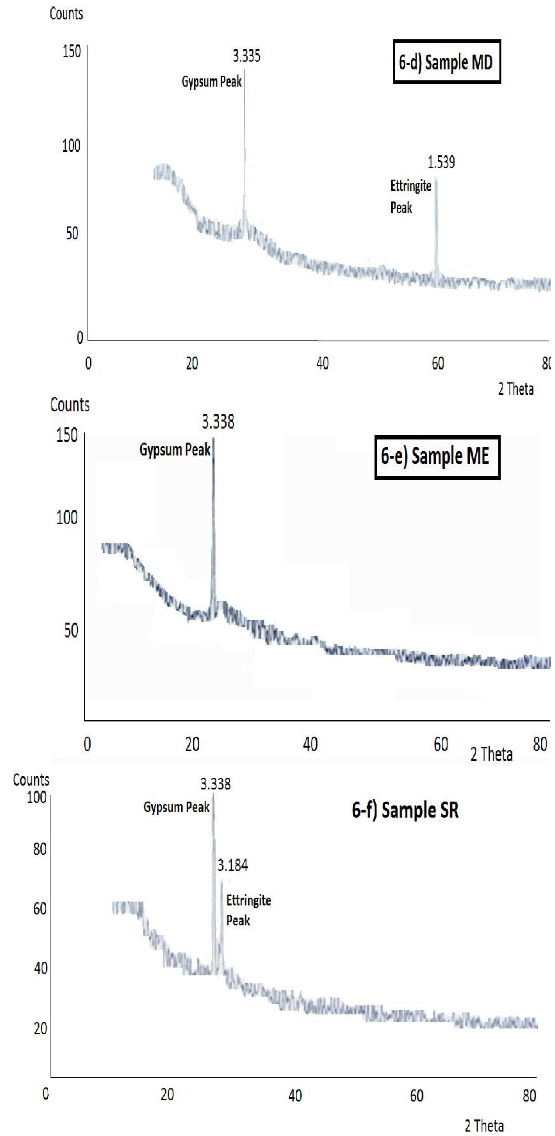


Figure 6 (a,b,c,d,e,f): XRD results of the Sulfate test showing the ettringite and gypsum formation

The results on the graphs of the XRD are largely in agreement with the results which were obtained in the case of expansion tests. In the figure 6-a the peaks are large and high then the other samples which are indicating that gypsum and ettringite had been formed in case of CEM II sample. The gypsum peak in the figure 6-a was shown strongly. The mortar prisms prepared using CEM-I (OPC) and 70% GGBS showed no ettringite peak as shown in the figure 6-e. The broad peak in the figure had been relating that poorly crystalline gypsum had been formed. The same behavior was observed by CEM-II (Portland Limestone cement). Both the mixes containing CEM-II and GGBS reacted very little and there were no obvious peaks as shown in figures 6-b and 6-c. These graphs are showing that ettringite had not been formed or forming at later stages but gypsum peaks were again visible. In the figure 6-f the peaks are showing that the layers of gypsum and ettringite had been formed in the case of sample prepared using Sulfate resisting Portland cement. So the results obtained from this test are indicating that GGBS containing samples perform better in the sulfate solutions than the other samples.

4. Sulfuric Acid Test

Mass Loss Results

The loss of mass of the concrete specimens was considered an acceptable means of assessing the performance of each of the mixes in a sulfuric acid environment. The results of this procedure indicate that there may be a slight increase in mass over the first twenty-eight days of exposure or very little mass loss. The concrete made from CEM II limestone cement with no addition of GGBS showed a higher initial gain in mass compared to the five other mixes shown in the figure 7, although the amount could be regarded as not significant. It has been indicated in figures 7. Although the figure indicates that 70% GGBS containing sample performed the best throughout the testing period. The mixes which performed the worst (SRPC) was considered to be not significant. This is the confirmation of the aggressive nature of the sulfuric acid solution and the inherent difficulties in exposing cementitious materials to this type of environment.

For both experiments there is very little difference between the performances of each of the six different mixes in terms of mass loss or compressive strength. The test results show that an addition of 70% GGBS improves the resistance to mass loss after four months. It is evident that a total loss of cohesion has occurred throughout the specimen proving that while much of the ongoing reactions are surface oriented there are more forces at work beneath the cube surface. Furthermore, there may be a useful relationship between the initial visibility of exposed aggregate and the underlying state of the concrete integrity exposed to acidic environments as the cube strength data revealed.

Compressive Strength Tests

A number of concrete cubes were prepared and cured for 28 days and then divided into two groups. Half of the cubes were dipped into sulfuric acid solution and the other was stored in water for 112 days. The compressive strengths of each of the mixes were then taken at twenty-eight, fifty-six, eighty-four and one hundred and twelve days. The strengths obtained showed different behavior. The cubes dipped in acid showed a totally different behavior as compared to the others dipped in water as explained in figure 8. The cubes dipped in acid showed a constant decrease in strength.

5. Summary of Results

The results of this investigation have clearly outlined the cause of concrete deterioration in wastewater treatment systems. Consequently, a clear distinction has been drawn between degradation due to sulfate attack and that due to a sulfuric acid attack in this environment. It is evident that neither the concrete standards nor concrete specifiers are taking into account the harsh nature of this form of attack by suitably distinguishing between the two corrosion phenomena. The laboratory programme has also failed to highlight a concrete specification that is capable of withstanding bio-deterioration. The range of aggressive environments associated with wastewater applications needs to be quantified and used as an input for future research work.

6. Conclusions

Sodium sulfate tests

The sodium sulfate experimental programme recorded an apparent relationship between expansion and time in days with each stage characterized by a specific physical deterioration mechanism. The main conclusions that can be drawn are thus:

i. The cement which performed worst under experimental setup was 100% CEM-I.
ii. CEM-II limestone cements were possessing and inherenting sulfate resisting capabilities that were superior to CEM -I cement. When combined with 50% or 70% GGBS it represented the best performing binder combination.
iii. The specimens which were containing GGBS performed very well than all other mixes regardless of the cement type.
iv. Deterioration was primarily due to bulging, spalling and warping, most likely as a result of the formation of gypsum. This type of mechanical deterioration is not generally expected in a wastewater environment.

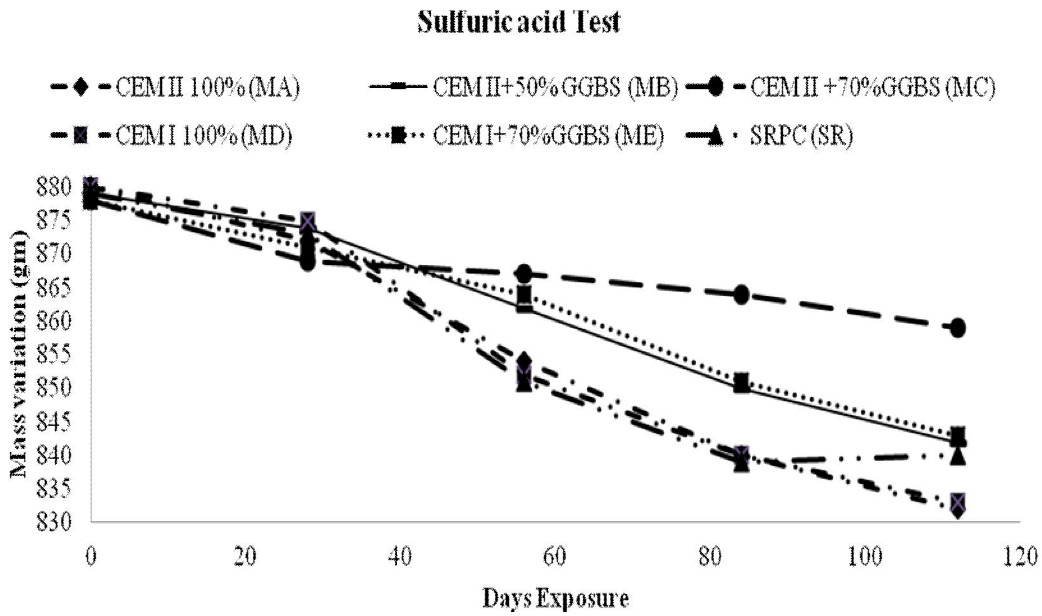


Figure 7: Mass loss of samples after sulfuric acid exposure

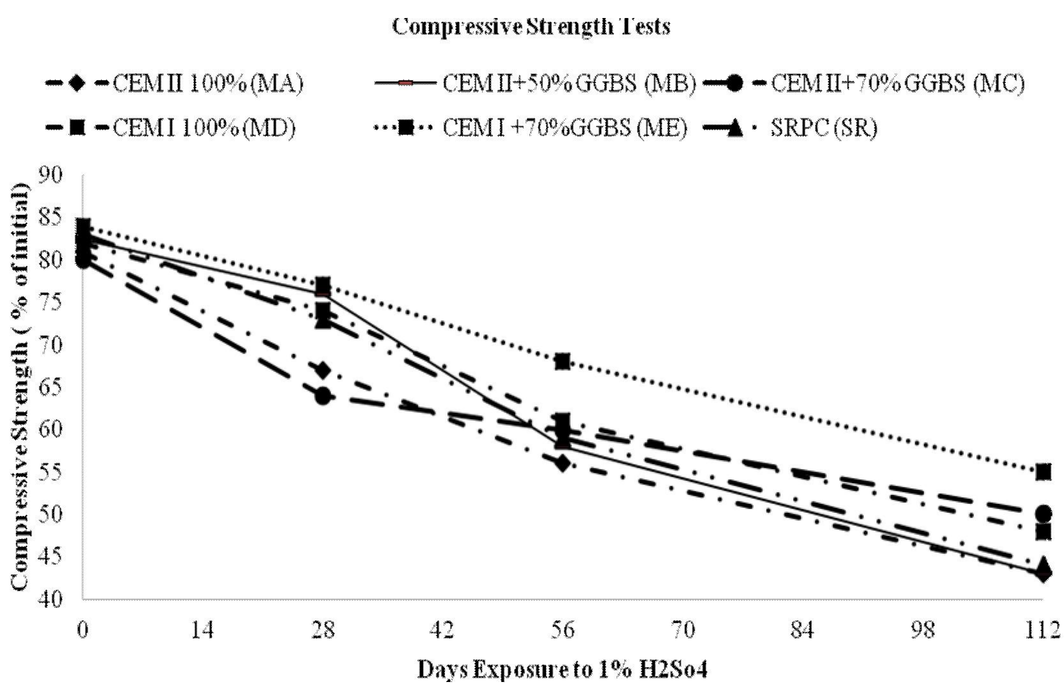


Figure 8: Influence of period of acid exposure on compressive strength

Sulfuric acid tests

The sulfuric acid test programme primarily indicated the inability of concrete to survive under very aggressive sulfuric acid solutions. Furthermore, a collaboration of existing data of acidic corrosion shows that this may apply to a large variety of acids including acetic and lactic acids. The findings show that solution pH may be a controlling force in the deterioration process. The test programme again served to highlight both the significant differences and slight similarities between sulfate and sulfuric acid based deterioration mechanisms. The main conclusions that can be drawn are thus:

i. Sulfate deterioration differs from that of sulfuric acid deterioration. Visual examination may confuse the two mechanisms because of the presence of gypsum to both.
ii. The 1% sulfuric acid solution (pH≈1.5) represents the most severe conditions to be expected in service. Actual pH levels may vary according to time, temperature and bacterial activity.
iii. The rate of visual deterioration of a 1% solution of sulfuric acid attack is more than that of a 5% sodium sulfate solution.
iv. Under this type of environment the use of GGBS and SRPC had no effect on the performance of concrete and also had little or no effect on improving resistance.
v. Mass loss may not be an accurate performance indicator of the deterioration level. Despite a difference between the cubes dipped in water and cubes dipped in sulfuric acid solution specimens, cube strengths revealed almost no change in performance.
vi. With reference to mass loss, there was very little distinction between the performances of each of the six mixes. Some minor differences were, however, noted. An initial increase (or no decrease) in mass for the first 28 days appeared to be common to all mixes and test conditions.
vii. The main deterioration mechanism consisted of the formation of gypsum on the external surfaces of the concrete specimens. This was followed by surface delamination and also some spalling.
viii. Sulfuric acid deterioration visually appeared to be more surface oriented than sulfate attack. The attack was concentrated primarily on the matrix of the cement.

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