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## Using concrete admixtures for sulphuric acid resistance

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**The degradation of concrete sewer pipes by sulphuric acid attack is a problem of global scope, resulting in substantial economic losses each year. In this study, five admixtures, which offer a range of potential improvement mechanisms, were used at various dosages to enhance the resistance of concrete made with Type 50E cement to chemical sulphuric acid attack. The resistance to sulphuric acid of concrete specimens incorporating these admixtures was measured and compared to that of control specimens. An attempt was made to determine whether there is a relationship between the effect of the various admixtures on mechanical strength and porosity and the resistance of concrete to H<sub>2</sub>SO<sub>4</sub> attack. Results indicate that metakaolin reduced the mass loss of concrete specimens due to immersion for eight weeks in H<sub>2</sub>SO<sub>4</sub> solutions having concentrations of 7% and 3% (by volume) by 38 and 25%, respectively, compared with that of the control specimens. Other admixtures (OCI, Caltite and Xypex) reduced the mass loss of concrete specimens in the range 12–20% for the 7% H<sub>2</sub>SO<sub>4</sub> solution and in the range 9–16% for the 3% H<sub>2</sub>SO<sub>4</sub> solution. Although silica fume effectively increased compressive strength and reduced the porosity of concrete, its contribution to the resistance of concrete to chemical sulphuric acid was minor. No clear relationship could be established between the mechanical and physical properties of concrete (compressive strength and porosity) and its resistance to sulphuric acid attack. It was also found that the decline in compressive strength of concrete specimens subjected to H<sub>2</sub>SO<sub>4</sub> attack was directly proportional to their mass loss, following a linear relationship.**

### 1. INTRODUCTION

The degradation of concrete sewer pipes and wastewater treatment facilities by sulphuric acid attack is a substantial challenge in North America and indeed worldwide, resulting in economic losses of several billions of dollars annually. A recent study in the United States estimated the associated annual cost at several billion US dollars.<sup>1</sup> The cost of damage to wastewater utilities alone was estimated at approximately US\$30 billion. A study of sewer systems in the city of Los Angeles estimated annual costs associated with the repair and replacement of collection systems damaged by sulphide corrosion at US\$200 million.<sup>2</sup> Kaempfer and Berndt<sup>3</sup> estimated the expenses of repair and maintenance of private and public sewage systems in Germany at about US\$100 billion, with 40% of the damage

attributed to corrosion by biogenic sulphuric acid attack. The US Environmental Protection Agency (EPA) undertook an extensive study of the extent of sulphuric acid corrosion in municipal wastewater collection and treatment systems and indicated that annual damage is in the order of tens of billions of dollars, a high price tag for a problem that has been known and studied for nearly a century.

Olmstead and Hamlin<sup>4</sup> were the first to report a rapid acid-related corrosion in concrete sewage pipelines. Hydrogen sulphide was recognised as a source of corrosion in Cairo's sewer pipe systems in 1920, where within 10 years the damage reached a depth of 5–9 in. (150 mm), nearly half of the thickness of the pipe wall.<sup>5</sup> It was initially believed that hydrogen sulphide formed by sulphate reduction in the sewage stream, then escaped into the sewer atmosphere to chemically oxidise and form sulphuric acid.<sup>6</sup> In 1945, Parker<sup>7</sup> identified *Thiobacillus* genus bacteria as the source of sulphuric acid formation at the crown of concrete sewer pipes. The most aggressive species of these bacteria is *Thiobacillus thiooxidans*, which was shown to grow well in the laboratory even when exposed to a 7% solution of sulphuric acid.<sup>8</sup>

The corrosion of concrete sewer pipes by sulphuric acid is initiated by sulphate-reducing bacteria (SRB), micro-organisms that reside in the slime layer of sewer pipes. These bacteria utilise sulphates as an oxygen source and release sulphide, which reacts with dissolved hydrogen in wastewater to form dissolved hydrogen sulphide and hydrosulphide ions. The dissolved hydrogen sulphide is then volatilised and released into the atmosphere of sewer pipes where it is oxidised into sulphuric acid by aerobic bacteria (*Thiobacillus*) that occupy the crown of sewer pipes.<sup>7</sup> Sulphuric acid is neutralised by reacting with the hydration products of the concrete matrix to form gypsum and ettringite. Both gypsum and ettringite possess little structural strength, yet they have larger volumes than the compounds they replace. This results in internal pressures, formation of cracks and eventually the loss of aggregates and thinning of the wall of the concrete pipes.<sup>9</sup>

### 2. ADMIXTURES FOR ENHANCING RESISTANCE OF CONCRETE TO H<sub>2</sub>SO<sub>4</sub> ATTACK

Previous attempts to improve the resistance of concrete to sulphuric acid attack focused on the use of CSA Type 50 (ASTM C150 Type V) cement and supplementary cementitious materials (SCM). Attiogbe and Rizkallah<sup>10</sup> reported that for prolonged

exposure periods, sulphate-resistant Portland cement (SRPC) does not appear to provide a better resistance to sulphuric acid attack than that provided by ordinary Portland cement (OPC). This is because sulphate attack is only one aspect of sulphuric acid attack on concrete. Fattuhi and Hughes<sup>11</sup> stated that concrete cubes made with OPC and SRPC, which were immersed in a channel containing a 3% solution of continuously flowing sulphuric acid, exhibited comparable mass losses. They attributed this behaviour to the large proportions of lime and calcium silicates forming in concrete made with both types of cement. Schmidt *et al.*<sup>12</sup> argued that conventional concretes produced using standard cements (Types I to V) are inadequate for preventing the degradation of concrete by sulphuric acid attack.

Torii and Kawamura<sup>13</sup> investigated the effect of using silica fume and fly ash as partial replacement for cement on the resistance of concrete to a 2% solution of sulphuric acid. They concluded that such a partial replacement for cement could not effectively prevent the acid-type deterioration involving surface scaling and softening of mortar. Jirasit *et al.*<sup>14</sup> suggested that concrete made with a cementitious materials content of 300 kg/m<sup>3</sup> (18.7 lb/ft<sup>3</sup>) and incorporating 50% fly ash as partial replacement for cement could resist a 3% H<sub>2</sub>SO<sub>4</sub> solution. On the other hand, Daczko *et al.*<sup>15</sup> argued that partial replacement of OPC by 8% silica fume could reduce the mass loss of concrete specimens immersed in an H<sub>2</sub>SO<sub>4</sub> solution with a pH of 1.0 by 30%. They also argued that using 8% metakaolin as partial replacement for OPC provided little contribution to the resistance of concrete to sulphuric acid attack. This paper investigates the effect of five different admixtures on the resistance of concrete to sulphuric acid attack, and attempts to relate such effects to mechanical and physical changes imparted by these admixtures. The relationship between the decline in compressive strength of concrete and its mass loss due to sulphuric acid attack was defined. Moderate reductions in mass loss of concrete due to the use of effective admixtures imply substantial gains in structural integrity and significant life cycle performance improvements, which may be of interest to manufacturers of sewer facilities and owners of water treatment infrastructure.

### 3. EXPERIMENTAL WORK

#### 3.1. Materials

Type 50E cement was used in the experimental programme to produce a total of 26 different concrete mixtures. Type 50E cement is a blended cement consisting of 65% OPC and 35% (by mass) of finely ground granulated blastfurnace slag. It complies with the requirements of CAN/CSA-A362-98 for blended hydraulic cement. Type 50E is primarily used to produce cement with properties and performance equivalent to those of CSA Type 50 Portland cement (ASTM C150 Type V). The chemical composition and some physical properties of Type 50E cement are listed in Table 1. Concrete specimens made with CSA Type 10 (ASTM C150 Type I) were also used in the test programme. Table 1 provides the chemical composition and some physical properties of CSA Type 10 Portland cement. The term 'control mixture' in the current study refers to concrete made with Type 50E cement.

Siliceous coarse aggregate with a maximum particle size of 14 mm (0.55 in.) and siliceous fine aggregate were used in producing all concrete mixtures. The specific gravity and water

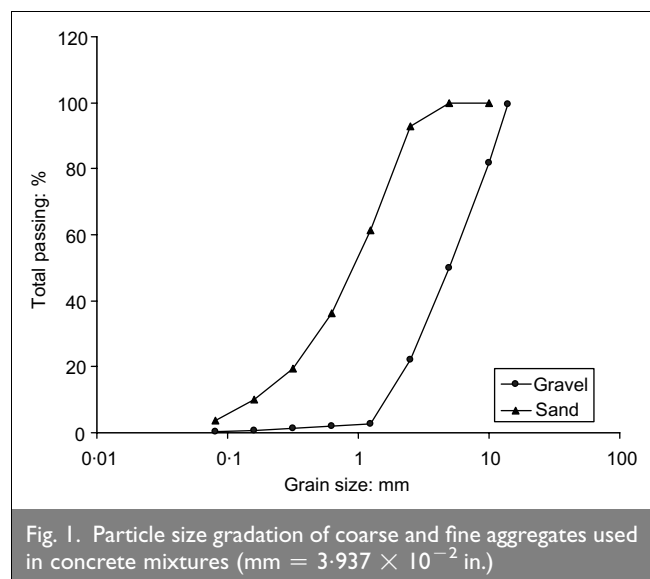
Component	Proportion: %	
	Type 50E	Type 10
SiO <sub>2</sub>	24.35	19.52
Al <sub>2</sub> O <sub>3</sub>	5.78	5.22
Fe <sub>2</sub> O <sub>3</sub>	2.07	2.42
CaO	55.74	61.34
MgO	4.48	2.47
SO <sub>3</sub>	3.14	4.09
K <sub>2</sub> O	0.13	1.21
Na <sub>2</sub> O	0.16	0.30
Loss on ignition	1.97	2.76
C <sub>3</sub> S	8.92	51.14
C <sub>2</sub> S	76.54	17.40
C <sub>3</sub> A	11.83	9.74
C <sub>4</sub> AF	6.29	7.35
Physical data:		
Blaine: m <sup>2</sup> /kg	399	412
Residue 45 μm: %	8.8	
Initial set: min.	140	180
Sulphate exp.: %	0.009	

Table 1. Chemical analysis and physical properties of Type 50E and Type 10 cements

absorption of the coarse aggregate were 2.68 and 0.8%, respectively. Those of the fine aggregate were 2.65 and 1.5%, respectively. The fineness modulus of the fine aggregate was 2.81. Fig. 1 shows the particle size distribution of the coarse and fine aggregates used in the study.

Five admixtures including silica fume, metakaolin, organic corrosion inhibitor (OCI), Caltite and Xypex were used in the experimental programme as listed below.

- Five levels of silica fume (6, 8, 10, 12 and 15% by mass of cement) and six levels of metakaolin (2, 4, 6, 8, 10 and 15% by mass of cement) were used as partial replacement for Type 50E cement.
- Five levels of OCI (3, 4, 5, 6 and 7 litres/m<sup>3</sup> of concrete) were used as an addition.
- Four levels of Caltite (20, 25, 30 and 35 litres/m<sup>3</sup> of concrete) were used as partial replacement for mixing water.
- Three levels of Xypex (1, 2 and 3% by mass of cement) were used as partial replacement for Type 50E cement.



Admixtures	Properties
Silica fume	Specific surface area = 30 m <sup>2</sup> /g; oversize, percentage retained on 45 µm, max = 5%; available alkalis as Na <sub>2</sub> O, max = 1.5%; loss on ignition, max = 6%; moisture content, max = 3.0%; SiO <sub>2</sub> , min = 85.0%
Metakaolin	Specific gravity = 2.6; bulk density = 0.4 g/cm <sup>3</sup> (25 lb/ft <sup>3</sup> ); SiO <sub>2</sub> = 52–54%; Al <sub>2</sub> O <sub>3</sub> = 42–44%; TiO <sub>2</sub> < 3.0%; Fe <sub>2</sub> O <sub>3</sub> < 1.4%; MgO < 0.1%; CaO = 0.1%; Na <sub>2</sub> O < 0.4%; K <sub>2</sub> O < 0.4%
OCI	Aqueous mixture of amines and esters; specific gravity = 0.98; pH = 10–12; meets ASTM C494
Calcite	A soluble ammonium stearate contains suspended microscopic asphalt particles; appearance: dark brown liquid; pH: 10.5 ± 1; specific gravity = 0.98 ± 0.02; volatiles (volume) = 78%; ammonium hydroxide (mass) = 1.3%; meets BS 1881-5 and BS 5075-2
Xypex	Dry powder composed of Portland cement, very fine treated silica, and various active chemicals; meets ASTM C267-77, C39-86, E329-95, C109, C266, and CRD C48-73

Table 2. Chemical and physical analyses of admixtures

Concrete components	Control mixture
Type 50E cement	430 kg/m <sup>3</sup>
Coarse aggregate	870 kg/m <sup>3</sup>
Fine aggregate	870 kg/m <sup>3</sup>
w/c ratio	0.35
Water	150 kg/m <sup>3</sup>
High-range water-reducing admixture	250 ml/100 kg
Slump	50 ± 10 mm

Table 3. Mixture proportions of control mixture

(e) Two levels of a binary Xypex–silica fume mixture—2% Xypex + 8% silica fume and 2% Xypex + 10% silica fume—were used as partial replacement for Type 50E cement.

The results of chemical and physical analyses of the five admixtures are provided in Table 2.

### 3.2. Concrete mixtures

The control mixture was produced using Type 50E cement. A water–cementitious materials ratio (w/cm) of 0.35 (typically used by concrete pipe producers of the Ontario Concrete Pipe Association) was used in all 26 mixtures. Mixture proportions of the control mixture (Table 3) were designed in accordance with the American method of selection of mix proportions (ACI 211.1). Mixture proportions of concrete specimens produced using Type 10 cement were similar to those of the control mixture (Type 50E cement). The other 25 mixtures were based on the control mixture but involved the use of various admixtures at different dosages. A high-range, water-reducing admixture was used to maintain the workability of all mixtures within the same range (slump of 50 ± 10 mm).

Fourteen 75 mm × 150 mm (3 in. × 6 in.) concrete cylinders from each of the 26 concrete mixtures were made and cured as per standard specifications (ASTM C192 and C172). Nine of the 14 specimens from each mixture were used to determine the compressive strength at 7, 28 and 120 days (three specimens tested at each date). Four specimens from each mixture were used for the sulphuric acid test programme. Small samples were taken from the remaining specimen for each mixture to carry out porosity tests.

All specimens were removed from the plastic moulds 1 day after casting and transferred to a curing room (relative humidity > 95%; temperature = 23 ± 1°C). The concrete specimens used for the porosity and sulphuric acid test programme were kept in the curing room for 28 days, whereas those used for the compressive strength tests were cured until testing.



Fig. 2. Set-up for sulphuric acid exposure tests

### 3.3. Sulphuric acid baths

Four polyvinyl chloride (PVC) containers with dimensions of 700 mm × 700 mm × 400 mm were constructed to carry out sulphuric acid tests (Fig. 2). Each pair of containers was connected by a 25 mm (1 in.) diameter PVC pipe and filled with sulphuric acid with a designated concentration. The first two containers contained 7% by volume of sulphuric acid (pH ≈ 0.3), whereas the remaining two containers contained 3% by volume of sulphuric acid (pH ≈ 0.6). The 7% solution represents the maximum concentration of sulphuric acid that can be generated by *Thiobacillus* bacteria. *Thiobacillus thiooxidans* bacteria were reported to be present at high counts in severely and moderately corroded sewers in Hamburg, Germany<sup>6</sup> and shown to grow in the laboratory in 7% sulphuric acid solutions.<sup>8</sup>

The two solutions of sulphuric acid were prepared by mixing concentrated sulphuric acid with a predetermined amount of distilled water. The sulphuric acid solution was circulated through each pair of acid baths using a multi-speed electrical pump with a flow rate of 0.3 litres/min (0.08 gallons/min). The circulation of sulphuric acid prevented stagnation and provided uniform conditions. The pH levels of the two sulphuric acid solutions were monitored daily using a portable digital pH meter (standard error ± 0.05). Concentrated sulphuric acid was periodically added to the solutions to maintain the pH levels within an acceptable range of the designated concentrations.

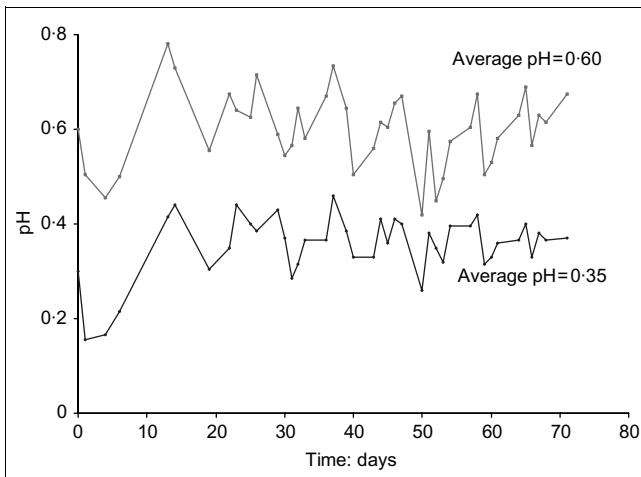


Fig. 3. pH readings for the two sulphuric acid solutions during period of immersion

Fig. 3 shows the daily readings of pH for the two solutions of sulphuric acid, which averaged 0.35 and 0.60, respectively for the entire test period.

As previously mentioned, four concrete specimens from each mixture were used for the sulphuric acid test programme, resulting in a total of 104 test specimens. The specimens were equally distributed among the four baths (26 specimens in each container) in such a manner that two specimens from each mixture were immersed in each of the two sulphuric acid solutions (pH of 0.3 and 0.6).

### 3.4. Sulphuric acid test measurements

At the end of the 28-day curing period for concrete specimens used in the sulphuric acid test programme, the specimens were oven dried at 105°C until constant mass, cooled at room temperature, weighed using an electronic scale (accuracy of  $\pm 0.01$  g) and then immersed into the sulphuric acid bath. It should be noted that wetting/drying can initiate degradation effects of its own and also exacerbate the effects of acid attack through cracking and other synergistic effects. Therefore, the results of this study should be interpreted in this context. Measurements were performed at 7, 16, 23, 30, 45 and 61 days of sulphuric acid immersion; at each date the specimens were removed from the acid solution, carefully rinsed with sulphuric acid and oven dried at 105°C until constant mass. The specimens were subsequently cooled at room temperature, weighed and then reimmersed into the sulphuric acid bath. The sulphuric acid test programme was terminated when the concrete specimens started losing coarse aggregates. Dissolved products that remained in the acid baths were collected, dried and subjected to sieve analysis to separate coarse aggregates. The mass of the coarse aggregates was found to account for 4.0 and 3.1% of the solid material lost by the specimens immersed in 7% and 3% sulphuric acid solutions, respectively, and thus was considered to present only a minor error in the measurements. The percentage of mass loss at each date was calculated according to the following equation

$$\text{Mass loss : \%} = \frac{M_1 - M_2}{M_1} \times 100$$

where  $M_1$  is the specimen's mass before immersion and  $M_2$  is the specimen's mass after immersion. It should be noted that mass

loss is commonly used to evaluate the deterioration of concrete under acid attack.<sup>10,11,16</sup> The direct exposure of concrete specimens to 3% and 7% sulphuric acid solutions accelerated the degradation of concrete specimens. It is important to note that *Thiobacillus* bacteria may take years to generate such concentrations of sulphuric acid. This mainly depends on the characteristics of the wastewater and hydraulic properties of the sewer pipe.<sup>17</sup>

### 3.5. Compressive strength measurements

Compressive strength tests were performed at 7, 28 and 120 days using a hydraulic press machine with a loading rate of 14.4 MPa/min (2.1 ksi/min) as per ASTM C39 guidelines. Three identical specimens from each mixture were tested at each date and the average value is reported. The compressive strength of concrete specimens after exposure to sulphuric acid solutions was also examined at the end of the 61-day immersion period to determine the relationship between the reduction in compressive strength of the corroded specimens and their mass loss. The reduction in compressive strength of the corroded specimens was calculated as follows (ASTM C267):

2	<p>Reduction in compressive strength: %</p> $= \frac{f'_{c_{28\text{-day}}} - f'_{c_{\text{after corrosion}}}}{f'_{c_{28\text{-day}}}} \times 100$
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where  $f'_{c_{28\text{-day}}}$  is compressive strength at 28 days and  $f'_{c_{\text{after corrosion}}}$  is compressive strength after 61 days of immersion in sulphuric acid.

### 3.6. Porosity measurements

Small samples (2 g) were taken from concrete specimens that were moist cured for 28 days. Porosity tests were carried out using the nitrogen adsorption method (ASTM D3037). The nitrogen adsorption technique used in this study has the ability to measure pores with a diameter between 0.3 and 300 nm.

All various tests were carried out at a room temperature of  $22 \pm 2^\circ\text{C}$ .

## 4. RESULTS AND DISCUSSION

### 4.1. Control mixture

The presence of 35% slag in Type 50E cement represents the main difference between Type 50E and Type 10 cements. Fig. 4 shows the progress in mass loss of the control specimens made with Type 50E cement plotted against time of immersion in sulphuric acid baths. The figure also shows the mass loss of concrete specimens made with OPC (Type 10 cement) and exposed to similar conditions, tested as part of an earlier study.<sup>17</sup> At the end of the test period, control specimens made with Type 50E cement experienced mass losses of 28.0 and 19.2% due to immersion in 7% and 3% sulphuric acid solutions, respectively. The corresponding mass losses of the control specimens made with Type 10 cement were 28.4 and 18.3%, respectively. Comparing the trend of mass loss against time for the two mixtures indicates that both concrete types deteriorated at similar rates, exhibiting comparable trends of mass loss against time in both concentrations of sulphuric acid. Thus, partial replacement of OPC with slag did little to enhance the resistance of concrete to attack by aggressive sulphuric acid solutions.



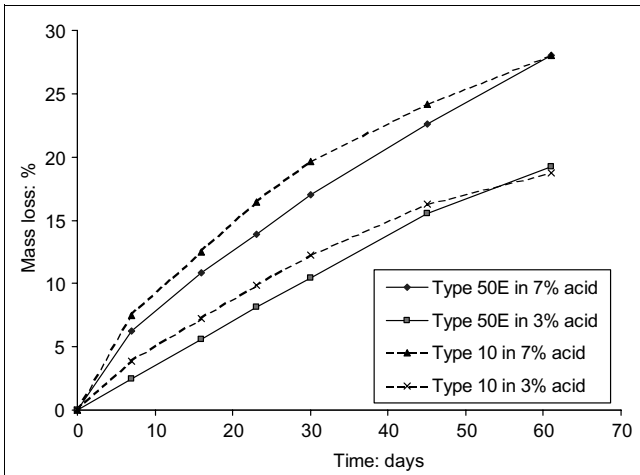


Fig. 4. Mass loss of concrete specimens made with Type 10 and Type 50E cements due to immersion for 61 days in 7% and 3%  $H_2SO_4$  solutions

#### 4.2. Effect of silica fume

The effect of using silica fume as partial replacement for Type 50E cement (which results in a ternary OPC–slag–silica fume binder) on the resistance of concrete to sulphuric acid attack is illustrated in Fig. 5. It can be observed that irrespective of the replacement dosage, the presence of silica fume in the concrete mixture had a minor effect on the resistance to the 7%  $H_2SO_4$  solution. For concrete specimens exposed to the 3%  $H_2SO_4$  solution, silica fume dosages less than 10% offered a marginal reduction in the mass loss of concrete specimens. The effect of using 15% silica fume as partial replacement for Type 50E cement on the rate and amount of mass loss during the 61-day test period is shown in Fig. 6. It can be seen that the rate of mass loss for the 15% silica-fume-modified specimens was similar to that of the control specimens. Furthermore, the amount of mass loss for the silica-fume-modified specimens was slightly higher than that of the control specimens in both acid solutions.

Silica fume, as will be explained later, significantly improved compressive strength and reduced porosity of concrete. Accordingly, it can be argued that the mechanisms, which improved compressive strength and reduced porosity, were not successful in improving the resistance of concrete to attack by

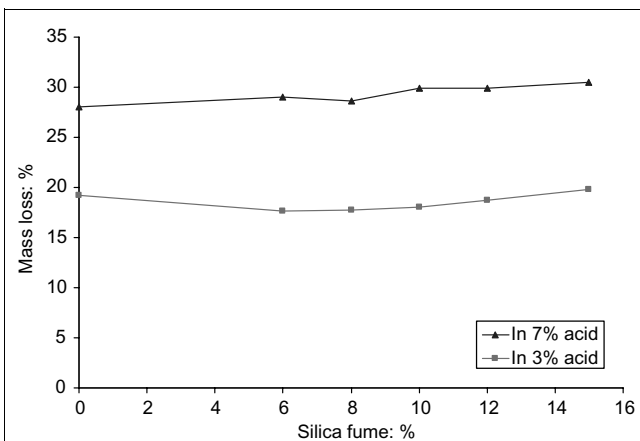


Fig. 5. Effect of silica fume as partial replacement for Type 50E cement on mass loss of concrete specimens immersed for 61 days in 7% and 3%  $H_2SO_4$  solutions

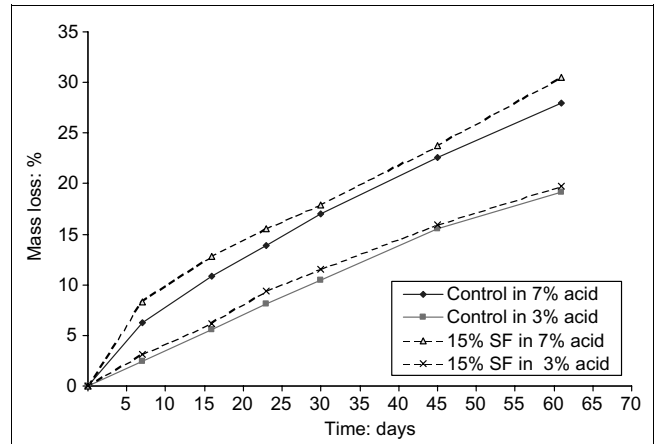


Fig. 6. Mass loss due to immersion in 7% and 3%  $H_2SO_4$  solutions plotted against time for control and 15% silica-fume-modified specimens

sulphuric acid having relatively high concentration. In other words, converting calcium hydroxide into calcium silicate hydrate and refining porosity of concrete did not improve its resistance to severe sulphuric acid attack. It was reported that CSH is vulnerable under attack by acidic solutions and dissolves to produce soluble salts.<sup>18</sup>

The findings of the present study are in contrast with those reported by Daczko *et al.*,<sup>15</sup> who found that partial replacement of OPC by 8% silica fume reduced the mass loss of concrete specimens immersed in  $H_2SO_4$ . This could be partially explained by the different concentrations of sulphuric acid used in the two studies. The lower concentrations of  $H_2SO_4$  (pH = 1.0, 2.5 and 4.0) used by Daczko *et al.* are less aggressive for CSH than those used in the current study (pH = 0.3 and 0.6). On the other hand, results for specimens incorporating silica fume in this study are in agreement with those reported by Torii and Kawamura,<sup>13</sup> who mentioned that both CH and CSH can be easily decomposed in a 2% sulphuric acid solution.

#### 4.3. Effect of metakaolin

Figure 7 shows the effect of various dosages of metakaolin on the mass loss of concrete specimens immersed for eight weeks in sulphuric acid solutions. A reduction of 38% and 25% in mass loss of concrete specimens due to immersion in sulphuric acid solutions with concentrations of 7% and 3%, respectively was achieved as a result of incorporating 15% metakaolin. The enhancement in resistance of concrete specimens to sulphuric acid was found to be proportional to the percentage of metakaolin and was more pronounced at the higher concentration of sulphuric acid (7%).

Figure 8 shows that the use of 15% metakaolin as partial replacement for Type 50E cement significantly reduced both the rate and amount of mass loss of concrete specimens subjected to sulphuric acid in comparison with that of control specimens. The figure also shows that no mass loss occurred during the first week of immersion in the 3%  $H_2SO_4$  solution for specimens incorporating metakaolin, suggesting that metakaolin had delayed the physical degradation of concrete. It is, however, important to note that the appearance of concrete specimens was altered during the first week of immersion, implying that some chemical activity was taking place at the concrete–acid interface.

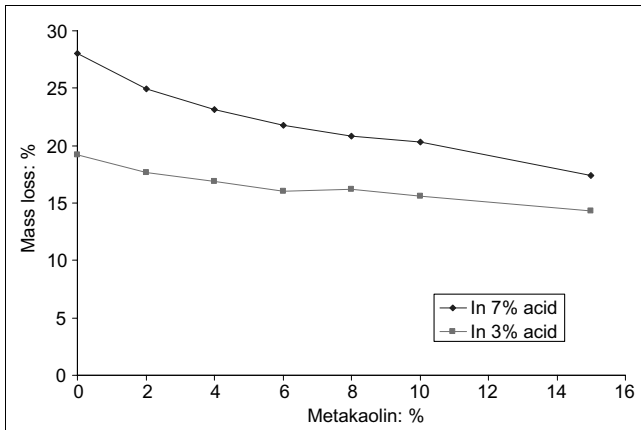


Fig. 7. Effect of various metakaolin dosages on mass loss of concrete specimens soaked for 61 days in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions

Although metakaolin and silica fume have comparable effects on the physical properties of hardened concrete, the chemical effect of metakaolin on the composition of the hydrated cement paste differs from that of silica fume. This is due to the creation of calcium aluminate hydrates (C<sub>2</sub>ASH<sub>8</sub>, C<sub>4</sub>AH<sub>13</sub>, C<sub>3</sub>AH<sub>6</sub>) in the metakaolin-modified concrete. It is believed that calcium aluminate hydrates may be more chemically stable in highly acidic environments than calcium silicate hydrates. This is indirectly supported by findings in the literature that high-alumina cements and calcium aluminate cements improve the resistance of concrete to sulphuric acid attack, possibly due to the high content of aluminates in these cements.<sup>2</sup>

#### 4.4. Effect of organic corrosion inhibitor

The organic corrosion inhibitor (OCI) used in this study consists of amines and fatty-acid esters. It is commonly used to provide protection of embedded steel reinforcement against corrosion and to improve the resistance of concrete to chemical attack. Nmai<sup>19</sup> suggested that the mechanism by which OCI inhibits corrosion and improves the resistance of concrete to chemical attack is a two-stage process. The first stage occurs during the mixing phase when the esters become hydrolysed to form carboxylic acid. Subsequently, the carboxylic anion is converted to insoluble calcium salt of the fatty acids that provides hydrophobic coating within the pores. This causes a reversed angle of contact whereby

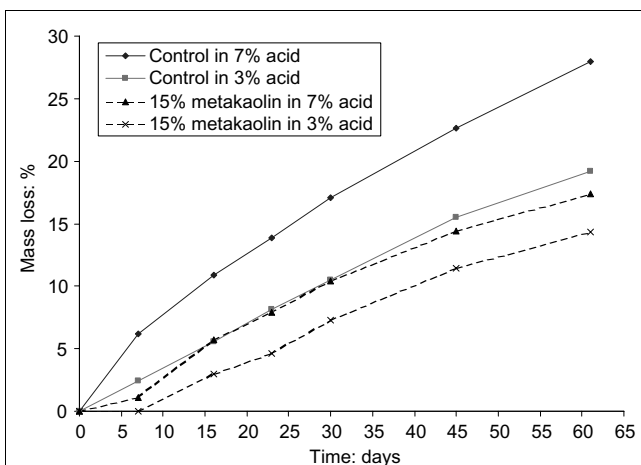


Fig. 8. Mass loss plotted against time of immersion for control and 15% metakaolin-modified concrete specimens

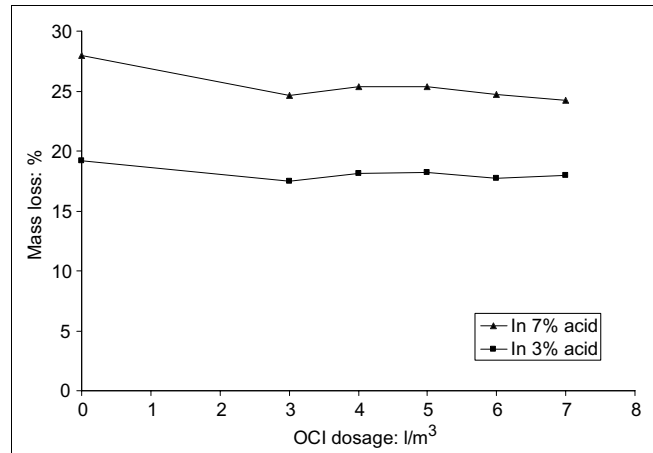


Fig. 9. Effect of different OCI dosages on mass loss of concrete specimens due to immersion for 61 days in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions

the surface tension forces push water out of the pores instead of into them. Due to the presence of the hydrophobic calcium salt lining, the diameter of pores is reduced, which in return results in a reduction in the aqueous species migration through the capillary pore matrix. The second stage occurs during placement of the concrete mixture when the film forming the amine component bonds with steel reinforcement to limit the availability of moisture, chloride and oxygen at the steel surface.

The mass loss of concrete specimens modified with different dosages of OCI due to immersion in sulphuric acid solutions is shown in Fig. 9. For both concentrations of sulphuric acid, mass loss of OCI-modified concrete specimens was somewhat lower than that of the control specimens. Fig. 10 shows mass loss plotted against time of immersion for control concrete specimens and specimens modified with 3 litres/m<sup>3</sup> (0.022 gallons/ft<sup>3</sup>) of OCI. It can be observed that the control and modified specimens underwent similar amounts of mass loss during the first month of immersion in either of the sulphuric acid solutions. During the second month of immersion, the rates of mass loss for the OCI-modified specimens were lower than those of the control specimens. Overall, a reduction in mass loss of 12% and 9% was achieved for OCI-modified specimens (3 litres/m<sup>3</sup>) immersed in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions, respectively.

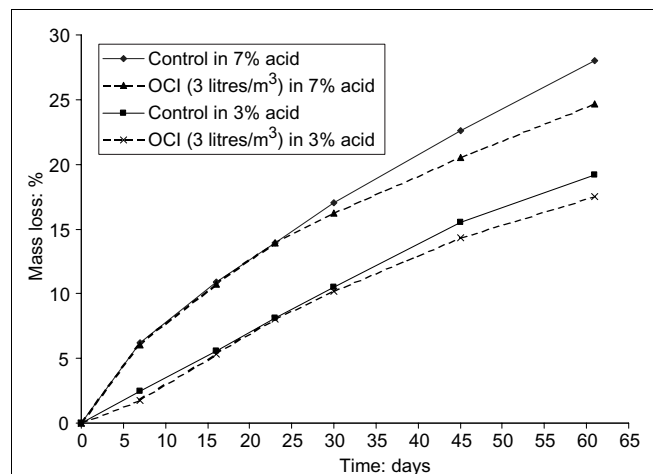


Fig. 10. Mass loss plotted against time of immersion for control and 3 litres/m<sup>3</sup> OCI-modified concrete specimens

While chemical reactions involving OCI that improve the resistance of concrete to acid are not fully understood, it is hypothesised that the role of OCI as a hydrophobic pore liner is a key contributor. This mechanism may reduce the amount of ion and water transport within the pore structure, thus reducing the reactions between  $H_2SO_4$  and cement hydration products. Furthermore, the presence of insoluble calcium salt of the fatty acids in the matrix of the OCI-modified concrete might also contribute to the enhanced resistance to sulphuric acid attack.

#### 4.5. Effect of Caltite

The main active ingredient of Caltite is soluble ammonium stearate, which represents 30% by weight. Caltite affects concrete properties chemically and physically. The chemical effect occurs between the ammonium stearate phase and calcium hydroxide in the cement paste, which produces calcium stearate and releases ammonia gas. Consequently, a water-repellent membrane coats the inside of capillary pores and reverses the capillary suction effect in concrete. The physical effect of Caltite occurs during the hardening stage, as suspended microscopic asphalted particles (polymer globules) present in the Caltite move with the bleed water, collecting in capillary voids. These particles can block capillaries when the hardened concrete is exposed to external moisture ingress.

The effect of various Caltite dosages (20–35 litres/ $m^3$  of concrete) on the mass loss of concrete specimens subjected to 61 days of immersion in sulphuric acid solutions is depicted in Fig. 11. A Caltite dosage of 20 litres/ $m^3$  (0.15 gallons/ $ft^3$ ) reduced the mass loss of concrete specimens subjected to 7% and 3%  $H_2SO_4$  solutions by 20% and 11%, respectively. Increasing the Caltite dosage beyond 20 litres/ $m^3$  (0.15 gallons/ $ft^3$ ) was found to provide no additional benefits. Mass loss against time of the control specimens and those modified with 20 litres/ $m^3$  (0.15 gallons/ $ft^3$ ) of Caltite are shown in Fig. 12. It can be observed that the presence of Caltite in concrete decreased both the absolute value and the rate of mass loss due to immersion in sulphuric acid solutions, especially for concrete specimens exposed to the 7%  $H_2SO_4$  solution.

The improved resistance of the Caltite-modified concrete to sulphuric acid attack can be attributed to the substitution of calcium hydroxide, a vulnerable compound to acid attack, by calcium stearate, a product that is relatively more stable in acidic

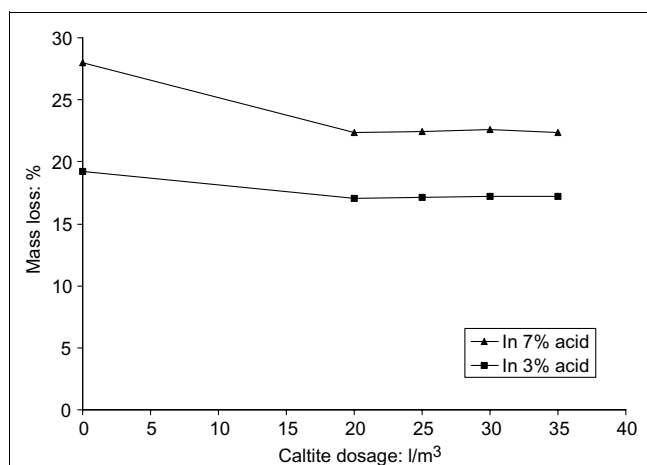


Fig. 11. Mass loss of concrete specimens modified with various Caltite dosages due to immersion for 61 days in 7% and 3%  $H_2SO_4$  solutions

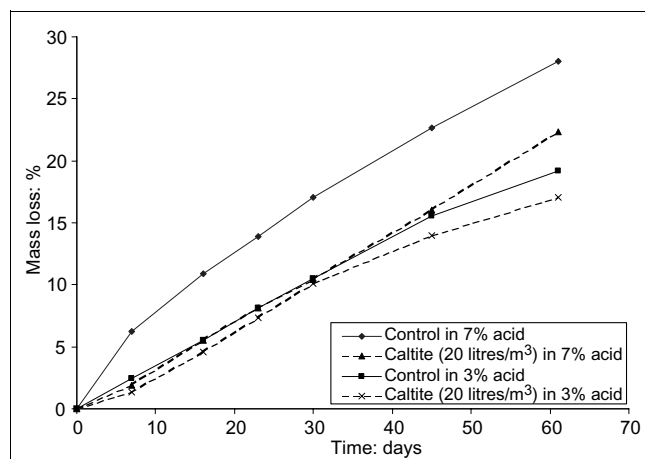


Fig. 12. Mass loss plotted against time of immersion for control concrete specimens and specimens modified with 20 litres/ $m^3$  of Caltite

environments. Another possible contributing factor is the hydrophobic action of Caltite, which might reduce the rate of chemical reactions between sulphuric acid and concrete hydration products by limiting the availability of ions and moisture. The presence of polymer globules in the capillary voids of the Caltite-modified concrete might provide additional resistance to the concrete against attack by sulphuric acid through a reduction of transport mechanisms.

#### 4.6. Effect of Xypex

Xypex is a dry powder composed of Portland cement, very fine treated silica and various active chemicals. By means of diffusion, the reactive chemicals in Xypex use water as a migrating medium to travel through the capillary tracts in concrete and react with moisture and cement hydration products (calcium hydroxide, mineral salts, mineral oxides, and unhydrated and partially hydrated cement particles). The result is a non-soluble crystalline structure that contributes to plugging pores and capillary tracts in concrete, thus reducing permeability.

Figure 13 shows the effect of three Xypex dosages on the mass loss of concrete specimens soaked for eight weeks in sulphuric acid solutions. It can be observed that concrete specimens modified with 1% and 2% Xypex exhibited slightly lower mass loss than that of the control specimens. This improvement was most significant for the 1% Xypex-modified specimens. The effect of using silica fume along with Xypex on the resistance of concrete specimens to sulphuric acid is also depicted in Fig. 13. For both acid concentrations, it is clear that the use of a silica fume-Xypex combination did not provide any additional improvement in acid resistance in comparison with that of concrete specimens modified with 2% Xypex.

Figure 14 shows the effect of using 1% Xypex as partial replacement for Type 50E cement on the mass loss of concrete specimens subjected to 7% and 3%  $H_2SO_4$  solutions. The mass loss appears to be divided into two regions. In the first region, ending at the third week of immersion, both the control and the 1% Xypex-modified specimens exhibited nearly identical rates of mass loss. Rates of mass loss for the modified specimens were lower than those of the control specimens in the second region

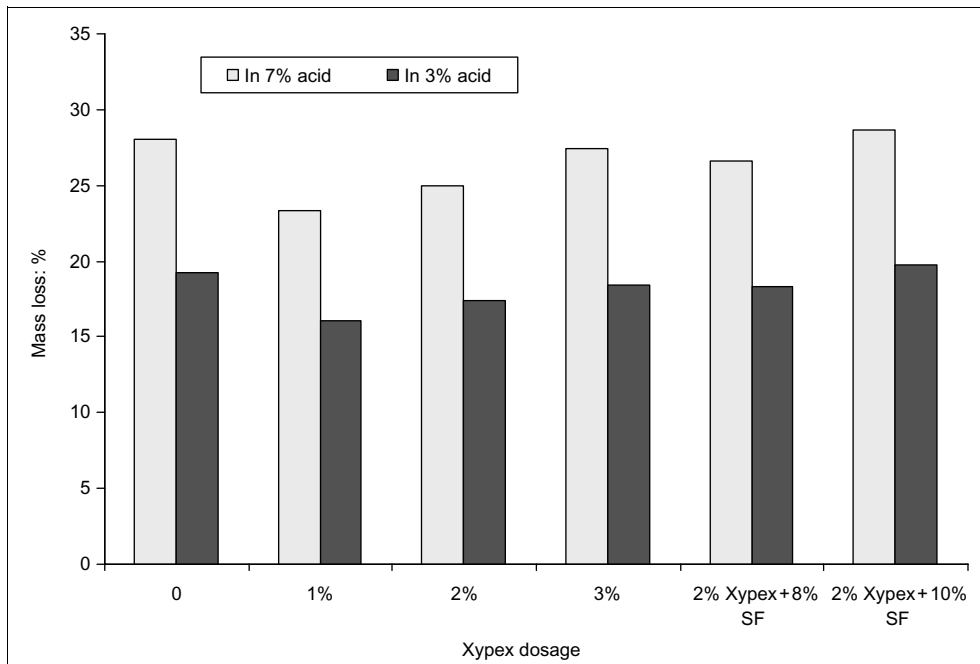


Fig. 13. Effect of Xypex and Xypex–silica fume combination on mass loss of concrete specimens due to immersion for 61 days in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions

formation of a denser cement paste–aggregate transition zone (ITZ, interfacial transition zone), refinement of microstructure and transforming CH into CSH through the pozzolanic reaction.

Similar to silica fume, metakaolin effectively improved the compressive strength of concrete at all ages. Using 15% metakaolin as partial replacement for Type 50E cement resulted in a compressive strength increase of 49, 27 and 22% at 7, 28 and 120 days, respectively. Due to its relatively high surface area and its chemical composition, metakaolin enhances the cement paste–aggregate ITZ and improves the strength and durability of concrete by replacing CH with CSH and CAH.

(between the fourth and the eighth week of immersion). The use of 1% Xypex decreased mass loss by 17% and 16% for concrete specimens immersed in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions, respectively, in comparison with that of the control specimens. This improvement is probably due to the formation of non-soluble crystalline products and the possible activation of the cement hydration process due to the presence of Xypex.

#### 4.7. Effect of admixtures on compressive strength

The effect of various dosages of the five admixtures considered in this study on the compressive strength of concrete specimens at ages 7, 28 and 120 days is provided in Table 4. It can be observed that the incorporation of silica fume in concrete mixtures significantly enhanced the compressive strength of concrete at all ages. A dosage of 15% silica fume resulted in an increase in the compressive strength of concrete specimens at 7, 28 and 120 days by 41, 18 and 14%, respectively. The contribution of silica fume to the compressive strength of concrete can be attributed to the

The effect of OCI on the compressive strength of concrete specimens at different ages was marginal. Whereas OCI dosages less than 5 litres/m<sup>3</sup> (0.037 gallons/ft<sup>3</sup>) somewhat increased compressive strength, dosages higher than 5 litres/m<sup>3</sup> (0.037 gallons/ft<sup>3</sup>) slightly decreased it. This is partially because OCI was used as an additive not as partial replacement for mixing water. The compressive strength of concrete specimens at different ages generally decreased with the use of Caltite. Xypex was used in small amounts (1–3%) therefore its effect on compressive strength was minimal. The use of a silica fume–Xypex combination effectively increased the compressive strength of concrete specimens, but this is mostly due to the effect of silica fume.

#### 4.8. Relationship between mass loss and 28-day compressive strength

The relationship between the 28-day compressive strength of concrete specimens and their mass loss due to immersion in 7% and 3% sulphuric acid solutions is shown in Fig. 15. Each graph in the figure is divided into four quadrants, with their point of intersection represented by the average 28-day compressive strength and mass loss of the control specimens. In both concentrations of sulphuric acid (Fig. 15(a) and (b)), points that represent concrete modified with various levels of metakaolin are located in the upper-left quadrant, which represents a performance of concrete having higher compressive strength and lower mass loss compared with that of the control mixture. Concrete modified with OCI or Xypex is generally located in the lower-left quadrant, which represents a performance of concrete having lower compressive strength and lower mass loss compared to that of the control mixture. The behaviour of Caltite- and silica-fume-modified concrete mixtures depended on the concentration of sulphuric acid. In the higher concentration solution (7% H<sub>2</sub>SO<sub>4</sub>), Caltite- and silica-fume-modified concrete specimens exhibited lower and higher mass loss, respectively compared with that of the control mixture. In the lower

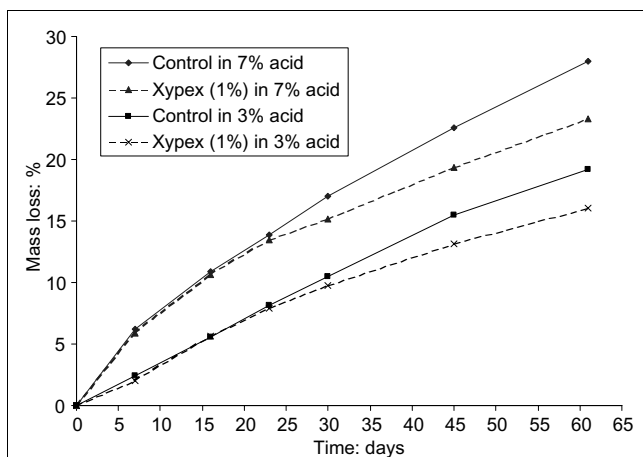


Fig. 14. Progress of mass loss of control and 1% Xypex-modified concrete specimens due to immersion for 61 days in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions



Concrete mixture	Compressive strength: MPa		
	7 days	28 days	120 days
Control mixture	40.2	57.6	61.7
Silica fume 6%	50.1	58.1	61.7
Silica fume 8%	50.5	60.6	62.4
Silica fume 10%	51.8	60.8	64.7
Silica fume 12%	54.3	62.2	65.6
Silica fume 15%	56.7	68.0	70.5
Metakaolin 2%	48.6	59.5	64.4
Metakaolin 4%	54.5	68.5	70.8
Metakaolin 6%	59.4	70.0	71.3
Metakaolin 8%	60.0	71.9	73.6
Metakaolin 10%	60.2	72.2	75.1
Metakaolin 15%	60.0	73.2	75.1
OCI 3 litres/m <sup>3</sup>	43.1	55.4	62.9
OCI 4 litres/m <sup>3</sup>	41.9	57.3	60.5
OCI 5 litres/m <sup>3</sup>	42.7	56.9	58.5
OCI 6 litres/m <sup>3</sup>	39.0	52.8	57.6
OCI 7 litres/m <sup>3</sup>	37.1	50.9	57.6
Caltite 20 litres/m <sup>3</sup>	38.9	51.0	55.0
Caltite 25 litres/m <sup>3</sup>	40.3	52.0	57.6
Caltite 30 litres/m <sup>3</sup>	44.1	53.6	58.8
Caltite 35 litres/m <sup>3</sup>	37.5	49.1	51.7
Xypex 1%	40.2	49.7	59.7
Xypex 2%	40.9	52.7	60.2
Xypex 3%	43.4	55.2	66.4
Xypex 2% + 8% silica fume	49.5	59.2	71.9
Xypex 2% + 10% silica fume	51.2	62.0	73.1

Table 4. Average compressive strength of control and modified concrete mixtures

concentration (3% H<sub>2</sub>SO<sub>4</sub>), three dosages of Caltite resulted in a higher mass loss, whereas four levels of silica fume marginally decreased the mass loss of concrete specimens compared with that of the control specimens.

These results suggest that the mechanisms responsible for improving the compressive strength of concrete are not necessarily the same as those that improve its resistance to sulphuric acid attack. Another observation is that the concentration of sulphuric acid had a significant effect on the performance of admixtures. For instance, five concrete mixtures modified with silica fume experienced a higher mass loss than that of the control mixture when exposed to a 7% sulphuric acid solution. The performance of the same specimens was different when exposed to a 3% sulphuric acid solution, since some of these mixtures exhibited a slightly lower mass loss than that of the control mixture.

#### 4.9. Effect of admixtures on porosity

The nitrogen adsorption technique used in this study has the ability to measure pore diameters between 0.3 and 300 nm. The nominal diameter of gel pores is about 3 nm, whereas capillary pores are one to two orders of magnitude larger.<sup>20</sup> These values vary depending on the water/cementitious materials (w/cm) ratio. In well-compacted concrete having low w/cm ratio, capillary pores are expected to be in the 10–50 nm range. Accordingly, results of the nitrogen adsorption technique cover both gel and capillary pores of the concrete mixtures, while entrapped air and capillary pores of diameter larger than 300 nm are not accounted

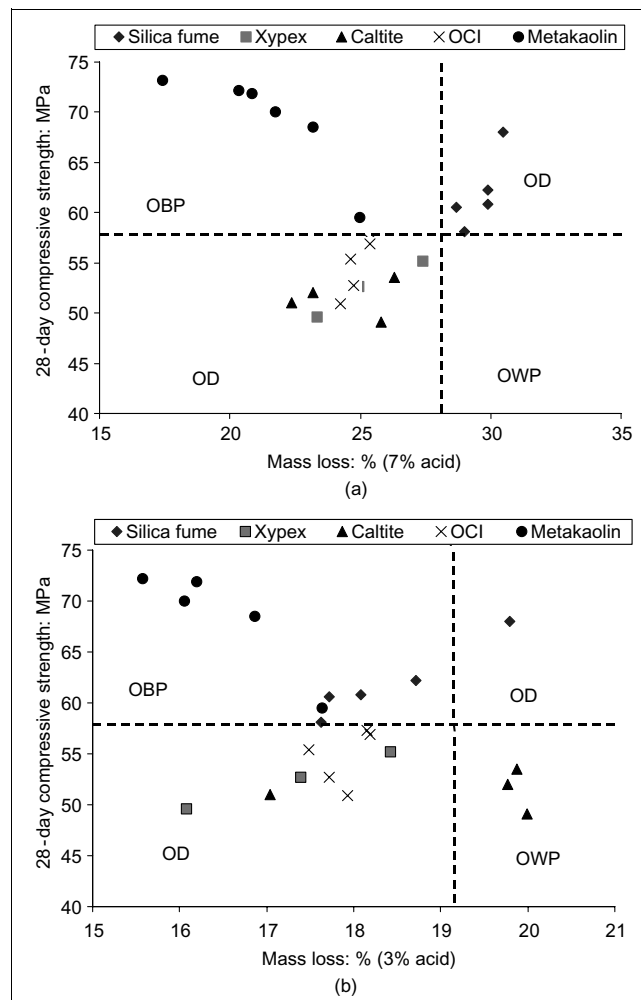


Fig. 15. Twenty-eight day compressive strength of concrete specimens plotted against mass loss due to immersion for 61 days in H<sub>2</sub>SO<sub>4</sub> with a concentration of (a) 7% and (b) 3%. OBP, overall better performance; OWP, overall worse performance; and OD, object dependent

for. The limitations of this technique and other techniques (e.g. mercury intrusion porosimetry) are beyond the scope of this paper and are discussed in detail elsewhere.<sup>21,22</sup>

The porosity of selected concrete specimens is summarised in Table 5. Admixtures including Caltite and Xypex slightly decreased the porosity of concrete specimens made with Type 50E cement. OCI somewhat increased porosity, while a dosage of 10% metakaolin reduced it by 38%. This level of reduction in porosity achieved by metakaolin is approximately similar to its effect on enhancing the resistance of concrete to sulphuric acid attack. Silica fume also effectively decreased the porosity of concrete specimens.

A review of test data, however, suggests that the relationship between the porosity of concrete and its resistance to sulphuric acid attack is not a consistent one. For instance, both silica fume and metakaolin reduced the porosity of concrete, but only metakaolin improved the resistance to acid attack. On the other hand, OCI improved the resistance of concrete to sulphuric acid attack while increasing its porosity. Thus, it can be argued that porosity alone cannot be used as a single indicator for establishing the resistance of concrete to severe sulphuric acid attack. The chemical composition and structure of hydration

Mixture	Porosity: cm <sup>3</sup> /gm
Control mixture	12.1 × 10 <sup>-3</sup>
Silica fume (12%)	9.3 × 10 <sup>-3</sup>
Metakaolin (10%)	7.5 × 10 <sup>-3</sup>
OCI (5 litres/m <sup>3</sup> )	14.2 × 10 <sup>-3</sup>
Caltite (30 litres/m <sup>3</sup> )	11.6 × 10 <sup>-3</sup>
Xypex (2%)	11.7 × 10 <sup>-3</sup>

Table 5. Nitrogen adsorption porosity values of control and modified concrete mixtures

products is a crucial parameter that could determine the relative vulnerability of the concrete matrix to degradation when exposed to a severe acidic environment, which is not captured by porosity measurements alone.

#### 4.10. Relationship between mass loss and compressive strength degradation

The relationship between the mass loss experienced by all concrete specimens subjected to sulphuric acid and the reduction in their 28-day compressive strength is shown in Fig. 16. It is observed that compressive strength declined as mass loss increased. This directly proportional relationship can be attributed to the fact that immersing concrete specimens in sulphuric acid results in loss of cement paste and its structural integrity, weakening of the concrete matrix, as well as a reduction in the specimen's diameter. A regression analysis of the data shown in Fig. 16 reveals a linear trend with an  $R^2$  value of 0.92. The best-fit line suggests that the expected percentile loss in compressive strength is approximately equal to 2.3 times the percentile mass loss, provided that the loss of coarse aggregates is minimal.

The potential benefit of an effective additive is demonstrated by considering the addition of 15% metakaolin as partial replacement for Type 50E cement, which was shown to reduce the mass loss of concrete specimens due to immersion in the 7% H<sub>2</sub>SO<sub>4</sub> solution by 38%. The net effect is an 87.40% (= 2.3 × 38%) decrease in the anticipated reduction in compressive strength. Thus, the useful life of precast concrete sewer pipes exposed to sulphuric acid attack could potentially be significantly enhanced by using admixtures that result in only moderate reduction in the mass loss due to sulphuric acid attack.

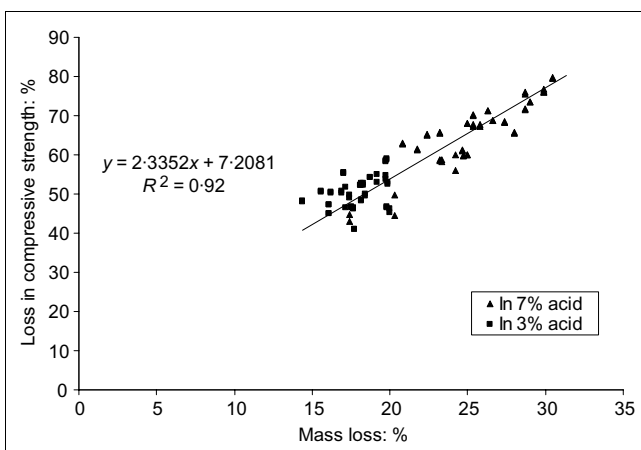


Fig. 16. Relationship between mass loss of concrete specimens and loss in their compressive strength due to 61 days of immersion in 7% and 3% H<sub>2</sub>SO<sub>4</sub> solutions

## 5. CONCLUSIONS

This study investigated the effect of various supplementary cementitious materials and admixtures on the resistance of concrete to sulphuric acid attack, its compressive strength and porosity. Based on the test data and analyses of results presented in this paper, the following conclusions can be drawn.

- The resistance of concrete made with Type 50E cement to degradation in an aggressive sulphuric acidic environment (7% and 3% H<sub>2</sub>SO<sub>4</sub>) was similar to that of concrete made with Type 10 cement. Hence, the presence of slag in Type 50E cement contributed little to the resistance of concrete to highly acidic environments.
- Although silica fume effectively improved the compressive strength and reduced porosity of concrete, its contribution to the resistance of concrete to severe sulphuric acid attack was minor.
- Metakaolin significantly enhanced the compressive strength and reduced porosity of concrete specimens made with Type 50E cement. A dosage of 15% metakaolin also decreased the mass loss of concrete specimens due to immersion for 61 days in sulphuric acid solutions with concentrations of 7% and 3% by 38% and 25%, respectively.
- The compressive strength at different ages and porosity of concrete were slightly affected by using various levels of OCI. However, OCI reduced the mass loss of concrete specimens due to 61 days of immersion in sulphuric acid solutions with concentrations of 7% and 3% by 12% and 9%, respectively.
- The compressive strength of Caltite-modified concrete was slightly lower than that of the control mixture, whereas the porosity of the control and Caltite-modified concrete specimens was comparable. Caltite reduced the mass loss of concrete specimens due to immersion for eight weeks in 7% and 3% solutions of sulphuric acid by 20% and 11%, respectively.
- Xypex slightly improved the compressive strength and reduced porosity of concrete. Its contribution to the sulphuric acid resistance of concrete specimens is indicated by reducing their mass loss due to 61 days of immersion in sulphuric acid solutions with concentration of 7% and 3% by 17% and 16%, respectively. Although a binary Xypex–silica fume admixture significantly enhanced compressive strength and reduced porosity of concrete, its effect on the resistance to sulphuric acid was limited.
- The mechanisms that contribute to higher compressive strength and/or lower porosity of concrete are not necessarily the same as those that enhance its resistance to sulphuric acid attack. For example, both silica fume and metakaolin were found to improve the compressive strength and reduce the porosity of concrete. However, only metakaolin enhanced the resistance of concrete to sulphuric acid attack.
- For all 104 concrete specimens exposed to different concentrations of sulphuric acid in this study, the resulting decline in compressive strength was found to be directly related to their mass loss ( $R^2 = 0.92$ ), following a linear relationship with a slope of 2.3. Thus, moderate reductions in mass loss achieved by admixtures can imply significant improvements in life-cycle performance of sewerage treatment facilities exposed to sulphuric acid. When concrete is exposed to a low concentration of sulphuric acid, mass loss may not be an adequate tool to measure the resistance of

concrete to H<sub>2</sub>SO<sub>4</sub>, and subsequently the mass loss–compressive strength relationship will not be applicable.

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