

# SULPHIDE, SULPHATE AND SULPHURIC ACID CORROSION OF CONCRETE IN LABORATORY TESTS

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## Abstract

Portland cement (PC) concrete is generally a highly durable structural material. Nevertheless, certain chemical actions and aggressive environments in a livestock building can cause deterioration and total collapses of structures have occurred long before they have reached their design life.

The sulphide and sulphate resistance of three replicates of eight different reinforced concrete mixes were investigated in a laboratory study in which one half of the 48 specimens were half submerged in a sodium sulphate solution (20,000 ppm  $\text{SO}_4^{2-}$ ) and also exposed to hydrogen sulphide gas (1,000 ppm  $\text{H}_2\text{S}$ ). The other half of the 48 specimens was subjected to hydrogen sulphide gas only. The mixes included PC concrete with W/CM ratios of 0.4 and 0.5 and six mixes with cement replacements of slag, fly ash or silica fume, all with water/cementitious material (W/CM) ratio of 0.4.

After 23 cycles of testing over about 36 months, the electrochemical potential results and visual inspection of the reinforcing bars indicate that the PC concrete with 0.5 W/CM ratio was the least resistant against steel corrosion. Corrosion of the concrete was more critical than that of the steel. All treatments containing silica fume performed much better than PC40. Treatments that contained fly ash performed worse than plain PC concrete with the same W/CM ratio. Sulphate resistant cement concrete was more resistant than Type 10 Portland cement concrete, in both sets of tests. In general the samples that were exposed to hydrogen sulphide and sulphate corroded at a higher rate than those exposed to the  $\text{H}_2\text{S}$  gas only.

In subsequent tests 6 of the 8 mixes were exposed to 7% sulphuric acid for about one year. Preliminary results indicate the greatest mass loss for the concrete with 0.5 W/CM ratio and very similar amount of loss for the 5 mixes with W/CM ratio of 0.4. The least amount of mass loss was experienced by the mix with sulphate resistant cement.

**Keywords:** concrete corrosion, livestock building, manure, hydrogen sulphide, sulphate, sulphuric acid

## Introduction

Reinforced concrete for the use in agricultural facilities often exposed to aggressive environment conditions. Temporary storage of liquid manure underneath barn floors produce corrosive agents generated from aerobic and anaerobic fermentation, causing premature corrosion of reinforcement steel and degradation of the concrete. The rehabilitation of reinforced concrete structures due to corroding steel reinforcing bar is quite expensive compared with the use of good quality concrete at the time of construction.

For Canadian climatic conditions, storage of manure for a period of six months, or even longer, is generally required so that manure spreading can be avoided during winter. Anaerobic fermentation produces several gases of which hydrogen sulphide is the most corrosive leading to rapid deterioration of concrete floors in barns (Frénay and Zilverberg, 1993). Sulphate-reducing bacteria, known to thrive in animal confinement buildings, generate sulphuric acid as the end product of their metabolism. The sulphuric acid strength has been measured in the laboratory as equivalent to 7% (by volume)  $\text{H}_2\text{SO}_4$  (Hewayde 2005).

High humidity, the concentration of various gases above the liquid manure and the continuous wetting of concrete floor slats are all contributing factors (Svennerstedt et al., 1999). As a

consequence of the concrete degradation in some instances slatted floor have deteriorated to the point of requiring replacement in less than five years. In some swine barns in Ontario a 50% loss of expected service life was reported, when regular concrete mixes were used. It is estimated that an average annual cost of depreciation on all structures is about \$250 million and about \$100 to 150 million is spent on repairs.

In order to improve the durability of concrete recent investigations have been made in an attempt to reduce the rate of deterioration by changing the concrete composition (De Belie et al. 1997; Idriss 2000; Jiang 2002; Berge and Verhardsson, 2002). This study involves concrete made with various combinations of Portland cement, slag, fly ash and silica fume, subjected to hydrogen sulphide gas and sulphate solution.

The experimental part of the project will be described in this paper. The results of this research will help finding the most cost-effective solution in Ontario for reducing concrete corrosion to a minimum and enhance the service life of reinforced concrete in livestock buildings there.

### Materials and Methods

The concrete cover quality is the most effective protection and first defense against concrete corrosion. Thus, the study looked at the comparable durability of eight different concrete mixes that are practical and cost effective alternatives in the construction of livestock buildings and manure storage structures. One of the mixes was Portland cement type 10 with water/cementing material (W/CM) ratio of 0.5, which is considered a commonly used mix in farm building structures. Common wisdom is to lower the W/CM ratio to increase durability. So the same cement type was used with a W/CM ratio of 0.4. However, lowering the W/CM ratio invariably means increasing the cement content of the mix thereby increasing the  $C_3A$  content of the hardened cement paste and thus decreasing the sulphate resistance.

On order to reduce the Portland cement content six other mixes with various supplementary cementing materials, like slag, fly ash and silica fume, were included. The use of supplementary cementing materials to replace a portion of the Portland cement in the mix contributes to the properties of the hardened concrete through hydraulic or pozzolanic activity. It reduces the concrete permeability, decreases the cement content, and the W/CM ratio. It also, in general, improves the resistance of the concrete to sulphate attack by lowering the  $C_3A$  content of the hardened cement paste.

Concrete used for the construction of liquid manure handling and storage structures is subjected to sulphides, sulphates and sulphuric acid. In some locations the concrete is submerged continuously, in other places it is submerged some of the time and some locations are never submerged. Field observations indicate that the latter two situations lead to more severe corrosion than the totally submerged condition.

To reach the objectives of the research a combination of laboratory research and numerical simulation was selected. The laboratory research consisted of accelerated corrosion testing of concrete specimens by subjecting them to hydrogen sulphide gas and a sulphate solution in high concentrations. One half of the specimens was partially immersed in sodium sulphate (20,000 ppm  $SO_4^{2-}$ ) and also subjected to hydrogen sulphide gas (1,000 ppm  $H_2S$ ). This condition was chosen to simulate units of construction that are partially submerged in liquid manure. The second set was subjected to hydrogen sulphide gas only, a condition that may occur for concrete permanently above the manure. Each set consisted of the eight different mixes. The sulphuric acid corrosion was measured on those six mixes not containing slag by applying 2 cm<sup>3</sup> of 7% sulphuric acid three times per week to disc-shaped specimens.

The corrosion of the concrete and of the embedded reinforcing steel was measured separately. For the purpose of the embedded steel corrosion study, all treatments were tested with the half-cell potential between the concrete surface and the reinforcing steel to define the corrosion state. The reinforcing steel was visually inspected at three stages of the sulphide/sulphate experiment.

For the purpose of the study on concrete corrosion due to sulphate and/or sulphide, the compressive strength of all mixes was measured at 28 days. The sulphate solutions, in which the mixes were partially immersed, were analyzed and their pH's measured to follow the leaching of the alkalis from the concrete paste and the effect of  $H_2S$  gas on the solution acidity and  $SO_4^{2-}$  concentration. The

actual corrosion damage to the concrete was measured using the volume loss of the concrete at the end of the test period. The corrosion of concrete is either caused by a direct chemical reaction of the corrosive agents with the concrete at its surface, or by reactions below the surface after diffusion of the corrosive gases or liquids into the concrete. To try to measure the extent of the latter the total sulphur profile was determined across the concrete cover thickness. Finally, the mineralogy of the concrete was studied using powder x-Ray test to confirm the nature of the chemical reactions that took place and to confirm that these were similar to those that happened in a field situation where a pig barn collapsed occurred due to corrosion of the concrete structure.

The concrete corrosion from exposure to sulphuric acid was measured by weight loss. That aspect of the study is in progress at the time of writing.

Liquid swine manure has sulphate concentrations in the order of 1500 to 2000 ppm. Table 1 lists the concrete requirements in Canadian concrete standards for four degrees of exposure to sulphate (Kosmatka et al. 2002).

**Table 1.** Requirements for concrete subjected to sulphate attack

Degree of exposure	Water-soluble sulphate in soil (%)	Sulphate in groundwater (mg/L)	Max. water-cementing materials ratio	Portland cement type to be used
Very severe	Over 2.0	Over 10,000	0.40	Type 50 <sup>1</sup> plus a pozzolanic admixture
Severe	0.20 to 2.0	1500 to 10,000	0.45	Type 50
Moderate	0.10 to 0.20	150 to 1500	0.50	Type 20 <sup>2</sup>
Negligible	Below 0.10	Below 150	No restriction	No restriction

<sup>1</sup> CSA A5 (eq. to ASTM C1157 type HS) <sup>2</sup> CSA A5 (eq. to ASTM C1157 type MS)

The replacement of 35% of type 10 cement by slag is considered sufficient to provide Type 50 equivalence. The relative improvement in sulphate resistance through the use of fly ash is greater for low cement content mixes and in high sulphate exposure. Class F fly ash is effective in improving the sulphate resistance of concrete when used at suitable replacement rates. The higher the calcium content of a Class C fly ash, the less likely it is that it will provide benefits with regard to sulphate resistance (A Publication of Lafarge Canada Inc.). Unfortunately, in Ontario the available fly ash is type C.

Another effective mix is the use of silica fume. Due to reduced permeability, silica fume cement provides excellent sulphate resistance to concrete. Recent research shows that ternary blends containing slag cement or fly ash, along with silica fume and Portland cement, can be effective for sulphate resistance concrete (A Publication of Lafarge Canada Inc.).

It is intended that the objectives of the research be attained by the combination of laboratory research and numerical simulation. The corrosion testing in the laboratory are accelerated tests using hydrogen sulphide gas, and a sodium sulphate solution as the only corrosive agents. Acceleration is achieved by using much higher concentration than those experienced in the field, 1,000 ppm H<sub>2</sub>S and 20,000 ppm SO<sub>4</sub><sup>2-</sup>.

Eight mixes, which could reasonably be used in the construction of liquid manure tanks, floors and slats, were examined for corrosion resistance. Concrete cylinders (100 mm diameter by 100 mm high) made of Portland cement, limestone, sand and water, each with a 10 mm diameter by 90 mm long reinforcing steel bar embedded in the center. Six replicates for each mix were made. In all mixes a superplasticizer (CATEXOL 1000 SP-MN) is used (625 ml/100 kg of cementitious material) to reduce the water requirements in concrete and attain the necessary workability without the use of excess water. Also an air-entraining admixture (CATEXOL A.E.260) was used in all mixes (50 ml/100 kg of cementitious material) to increase concrete durability, improve workability and reduce bleeding. A further five larger replicates of each mix, 100 mm diameter and 200 mm height, were cast without a steel bar for compressive strength determination at 28 days (three replicates) and for the sulphuric acid corrosion tests. The mix proportions and materials used for all eight mixes are provided in Table 2. The coarse aggregate was crushed limestone.

**Table 2.** Mix proportions for all eight mixes

	PC50	PC40	SR	SC	SFC	FAC	SSFC	FASF
	PC with W/CM ratio 0.5	PC with W/CM ratio 0.4	Sulphate resisting cement	Slag cement	Silica fume cement	Fly ash cement	Silica fume & slag cement	Silica fume & fly ash cement
Cement type	10	10	50	10	10	10	10	10
Cement (kg/m <sup>3</sup> of concrete)	340	425	425	276	391	319	293	293
Water (kg/m <sup>3</sup> of concrete)	170	170	170	170	170	170	170	170
W/CM ratio	0.5	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Additive (% of cementitious material content)	---	---	---	35% slag	8% silica fume	25% fly ash	25% slag 6% silica fume	25% fly ash 6% silica fume

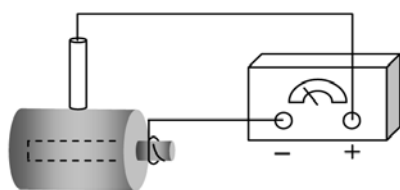
The top of all specimens, except those for the compressive strength tests, and the exposed ends of the steel bars were coated with an epoxy coating (TRU-GLAZE 4508 Chemical Resistant Epoxy Coating 4508-1000A and 4508-9999B with a ratio of 1:1), in order to prevent the diffusion of the corrosive ions through that surface.

For the sulphide/sulphate corrosion study the 48 specimens from 6 replicates were divided into two sets, each set had three replicates. The first set (i.e. 24 specimens) was tested partially (50%) immersed in sodium sulphate (20,000 ppm SO<sub>4</sub><sup>2-</sup>) and at the same time subjected to hydrogen sulphide gas (1,000 ppm H<sub>2</sub>S) above the surface of the sodium sulphate solution. Each treatment was submerged in a separate container, and placed in the upper level of a two-storey test chamber. The second set (i.e. the remaining 24 specimens) was subjected only to the hydrogen sulphide gas, nitrogen and air (0.1% H<sub>2</sub>S, 9.9% N<sub>2</sub>, and 90% air) in the lower level of the same chamber.

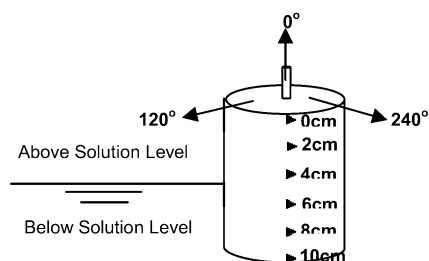
To keep the gas in the sealed Plexiglas test chamber at the required concentration a control circuit consisting of H<sub>2</sub>S sensor, solenoid valves, flow meter, control program and a gas cylinder (1% H<sub>2</sub>S, 99% N<sub>2</sub>) was used. The hydrogen sulphide gas cylinder lasted about 3 weeks, keeping the concentration of the gas inside the chamber at 1000 ppm. After this the chamber was left closed for one more week in order to lower the H<sub>2</sub>S concentration back to 0 ppm. Then all specimens were taken out of the chamber for the half-cell potential measurement, and thus they were subjected to air for another week. This test cycle was repeated approximately every five weeks.

The specimens for the sulphuric acid tests were 25 mm thick discs cut from the 100 mm diameter by 200 mm high unreinforced cylinders described earlier. Six mixes that did not include fly ash were included in these tests. The reason for excluding the fly ash containing mixes was that the type C fly ash that was used is not a viable solution for corrosion resistance in an acidic environment.

Standard 28-day compressive strength test were carried out for the 100 mm diameter by 200 mm high cylindrical specimens. The splitting tensile strength test (CSA A23.2-13C 1994) was carried out after 11, 15 and 23 cycles of exposure to the corrosive environment. This allowed the removal of the reinforcing bar for inspection of corrosion and provided a rough indication of the specimen's strength about 26, 32 and 41 months after the specimens were cast and after the exposure to a corrosive environment for most of that period.



**Figure 1** Half-cell potential measurement



**Figure 2** Positions where the potential measurements were taken

The half-cell potential,  $E_{\text{corr}}$ , was used to define the corrosion state of reinforcement bars. A copper-copper sulphate electrode (CSE) and a high impedance voltmeter were used to read  $E_{\text{corr}}$ . The half-cell potential measurement connection is shown in Fig. 1. The first half-cell potential measurements were taken after three cycles of exposure to the corrosive environment. Measurements were made every test cycle of about 5 weeks thereafter until the end of the experiment. Six potential readings were taken every 20 mm (at the air end, 20, 40, 60, 80, and 100 mm) along three meridians at 0°, 120°, and 240°, for a total of 18 readings per specimen (Fig. 2). The 18 readings were averaged. Corrosion potential measurements provide an indication of the oxidizing power of the environment in which a specimen is exposed.

At the end of the test period (after the 23<sup>rd</sup> test cycle), specimens were split open and concrete cover was removed to inspect the steel in the specimens. A visual assessment of the corrosion of reinforcing steel was performed. The corrosion was rated as three corrosion levels. Level 1 represents very slight or no visible rust on the surface. Level 2 shows a little rust at the surface. Level 3 indicates relatively heavy corrosion.

After the exposure of concrete specimens to the sulphate solution and/or sulphide gas, for the study period, the last set of specimens were air dried and brushed to remove all loose material. The volume loss of each specimen was determined using the water displacement method.

X-Ray powder diffraction patterns provided information on the phase, chemical and crystal structure. For the XRD test, 50 mg samples of the concrete paste were obtained from the outer surface of each mix and were well-ground to a uniform particle size to below 10  $\mu\text{m}$ . The test was carried out for three concrete mixes: PC40, SR, and SFC. The samples were taken from the last set of replicates for both corrosive environment exposures, a total of six samples. For the ones that were submerged the samples were taken from above the solution level. Also, samples were taken from a barn that collapsed in Innerkip in 2001, reportedly as a result of corrosion of the supporting piers. One sample was taken from the surface of a beam that supported a solid slab over the manure pit. Another sample was obtained from the outer surface of one of the piers that caused the failure.

The sulphuric acid corrosion tests were carried out on 25 mm thick by 100 mm diameter discs positioned on the flat. The present tests use discs that have the top surface of the cylinders from which they were cut on top, the cut surface on the bottom. Three times per week 2  $\text{cm}^3$  of 7% (by volume)  $\text{H}_2\text{SO}_4$  is dripped slowly on the surface of the discs. After each 10 applications (about 3 weeks) the discs are washed, scrubbed to remove loose material, dried in an oven for one week, and weighed. The weight losses are used as a comparative measure of corrosion.

## Results and Discussion

The compressive 28-day strengths of the specimens are shown in Table 3. The PC50 mix with W/CM ratio of 0.5 had much lower strength than the other seven mixes with W/CM ratio of 0.4. Of those the silica fume concrete, SFC, had the highest strength of 49.1 Mpa.

**Table 3.** 28-day compressive strength for the eight mixes

Mix	PC50	PC40	SR	SC	SFC	FAC	SSFC	FASF
W/CM ratio	0.5	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Compressive Strength (MPa)	34.0	48.6	47.4	42.3	49.1	47.0	43.5	46.0

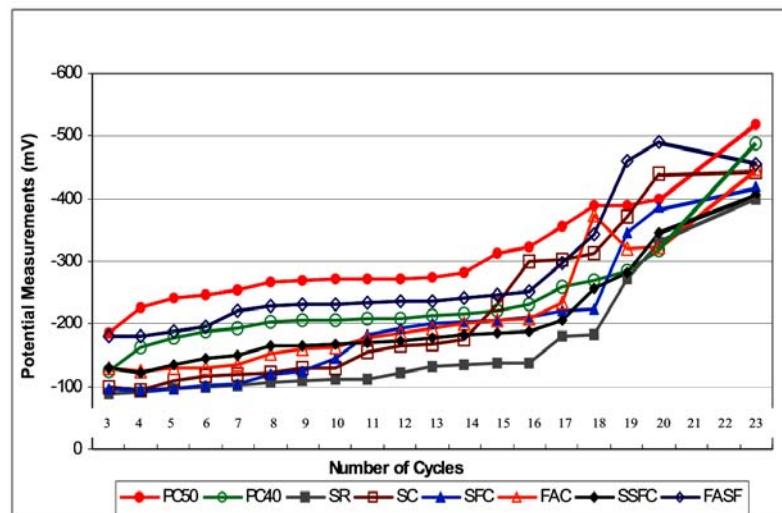
The splitting strengths were variable. The specimens exposed to H<sub>2</sub>S only gained some strength, about 1 kPa on average, between cycles 11 and 15, but lost about the same amount of strength between cycles 15 and 23. The specimens exposed to both H<sub>2</sub>S and sulphate gained similar amounts of tensile strength in the first part of the testing period but retained these strength until the end of testing, despite the loss of concrete volume to be discussed later.

The electrochemical potentials for the different mixes have been taken 19 times since the start of the experiment. They are plotted in Fig. 3 for the 8 mixes exposed to hydrogen sulphide and half submerged in sulphate solution. Fig. 4 presents the results for the 8 mixes exposed to hydrogen sulphide only. In examining the curves it should be noted that a potentials of  $-350$  mV may be considered a 90% probability of active steel corrosion. All values are the mean of all available replicates.

In Fig. 3 the potential measurements of PC50 were consistently the highest. It crossed the  $-350$  mV boundary at cycle 17. The values measured for the sulphate resistant concrete mix, SR were almost consistently the lowest, indicating excellent protection against steel corrosion.

They did not cross the  $-350$  mV till near the end of testing. All other mixes performed in between; mix FASF crossed the  $-350$  mV value at cycle 18, SC between cycles 18 and 19, mix SFC at about cycle 19 and SSFC at cycle 20.

The results in Fig. 4 indicate that the specimens exposed to H<sub>2</sub>S only experienced far less steel corrosion than did those exposed to both sulphates and H<sub>2</sub>S. Potentials were roughly one half. Indeed, none of the former reached the  $-350$  mV value. But as before, the PC50 mix exhibited the highest values almost consistently. Mix SSFC shows the lowest values up to cycle 18 after which mix SR gave the lowest value. In all mixes except PC50 there appeared to be a distinct increase in the rate at cycle 16.

**Fig. 3** Electrochemical potential of mixes exposed to sulphate and H<sub>2</sub>S

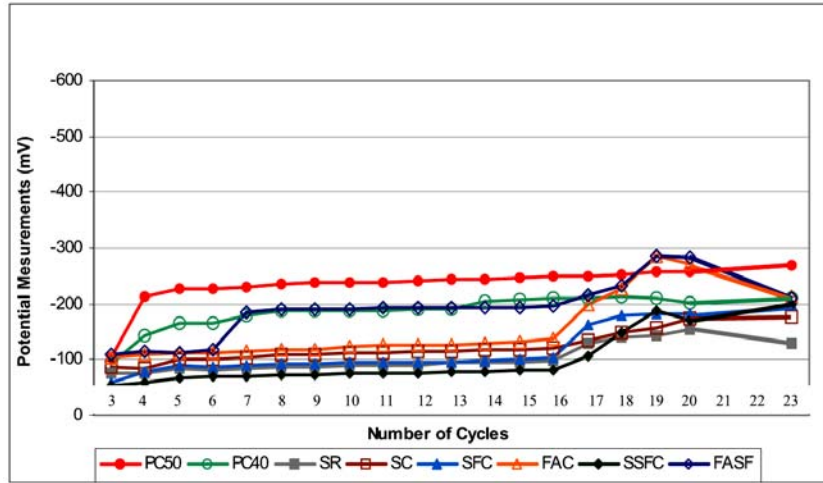


Fig. 4 Electrochemical potential of mixes exposed to H<sub>2</sub>S only

The degree of corrosion of the reinforcing steel was evaluated visually using numerical rating system of 1 to 3, 1 indicating no evidence of visible rust on the surface, and 3 indicating heavy corrosion. Table 4 presents the visual rating of steel surface corrosion of the 16 steel bars for the last set of specimens after the 23rd test cycle. The corrosion of the steel reinforcement was quite uniform in the specimens exposed to H<sub>2</sub>S only, regardless of the mix. All had medium amount of corrosion except mix FASF which had severe corrosion. There was considerable variability in the mixes exposed to H<sub>2</sub>S and sulphate. Mix SR had visibly the least amount of steel corrosion whereas in mixes PC50, PC40 and FAC the steel was heavily corroded. The visual corrosion ratings are in reasonable agreement with the electrochemical potential results.

**Table 4.** Visual rating of steel corrosion

Mix	H <sub>2</sub> S and sulphate	H <sub>2</sub> S only
PC50	3	2
PC40	3	2
SR	1	2
SC	2	2.5
SFC	2	2
FAC	3	2.5
SSFC	1.5	2
FASF	2	3

The volume loss experienced in the concrete because of corrosion is illustrated in Fig. 5. All concrete specimens exposed to sulphate solution and hydrogen sulphide gas showed higher volume loss than those exposed to hydrogen sulphide gas only. The volume loss was the greatest in mix PC40 exposed to both H<sub>2</sub>S and sulphate. The least amount of concrete lost to corrosion was in mix SR; SFC was second lowest.

Photographs of all specimens that experienced the full 23-cycle 3-year exposure to sulphate and/or sulphide are shown in Figs. 6a and 6b. Again, it is obvious from the photographs that all concrete specimens exposed to sulphate solution and hydrogen sulphide gas showed higher volume loss than those exposed to hydrogen sulphide gas only. And it is also obvious that most of the

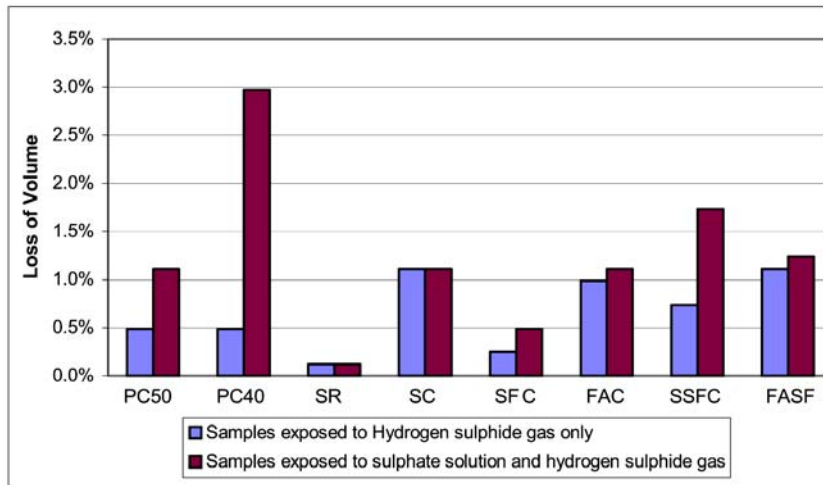


Fig. 5. Loss of volume for all mixes after 23 cycles of testing

concrete loss is located above the sulphate solution level. As was discussed earlier, the evaporation of the water from the concrete surface that contains high amount of alkali sulphates causes crystallization of salts, which in turn generates a disruptive pressure (Skalny et al. 2002). This is in good agreement with field observations of the piers partially submerged in liquid manure in the barn that collapsed in 2001 as a result of corrosion, after only 12 years of service.

X-ray diffraction results were obtained from surface scrapings from mixes PC40, SR and SFC exposed to  $H_2S$  only and those exposed to both  $H_2S$  and sulphate after 23 cycles of exposure. The scrapings were taken above the level of the sulphate solution in the case of the latter three specimens. Scrapings from the surfaces of these six specimens contained gypsum, but no ettringite. Samples taken from the piers and beams over the manure pit of the barn that collapsed in 2001 as a result of corrosion, after only 12 years of service, showed very similar diffraction patterns; again gypsum was the predominant corrosion product. As gypsum is the expected corrosion product at sulphate concentrations over 8,000 ppm it is clear that very high concentrations of sulphate (over 8,000 ppm) do occur in parts of manure structures.

After 110 applications of  $H_2SO_4$  over a period of approximately one year the PC50 experienced the greatest loss in mass of 5.2% of the original dry mass. The 5 specimens with a W/CM ratio of 0.4 had losses ranging from 4.0 to 4.7%. The least amount of mass loss was experienced by the sulphate resistant cement, SR. These preliminary results are not conclusive. What is evident from visual observation of the specimens that the limestone aggregate helps to reduce concrete deterioration from cement paste loss only and breakdown of the concrete structure. After a year of testing no pieces of coarse aggregate have become dislodged.

### Conclusions and Recommendations

After 23 cycles of exposure to sulphate and/or sulphide over about 36 months, the electrochemical potential results and visual inspection of the reinforcing bars indicate that the PC concrete with 0.5 W/CM ratio (PC50) is the least resistant to steel corrosion, as might be expected. All treatments containing silica fume are performing much better than PC40. Treatment FAC, and FASF stand out as performing worse than plain PC concrete with the same W/CM ratio confirming the fact that Class C fly ash, most commonly available in Ontario, does not provide improvement in sulphate resistance. SR was more resistant than Type 10 Portland cement, in both sets of tests. In general the samples that were exposed to hydrogen sulphide and sulphate corroded at a higher rate than those exposed to the  $H_2S$  gas only.



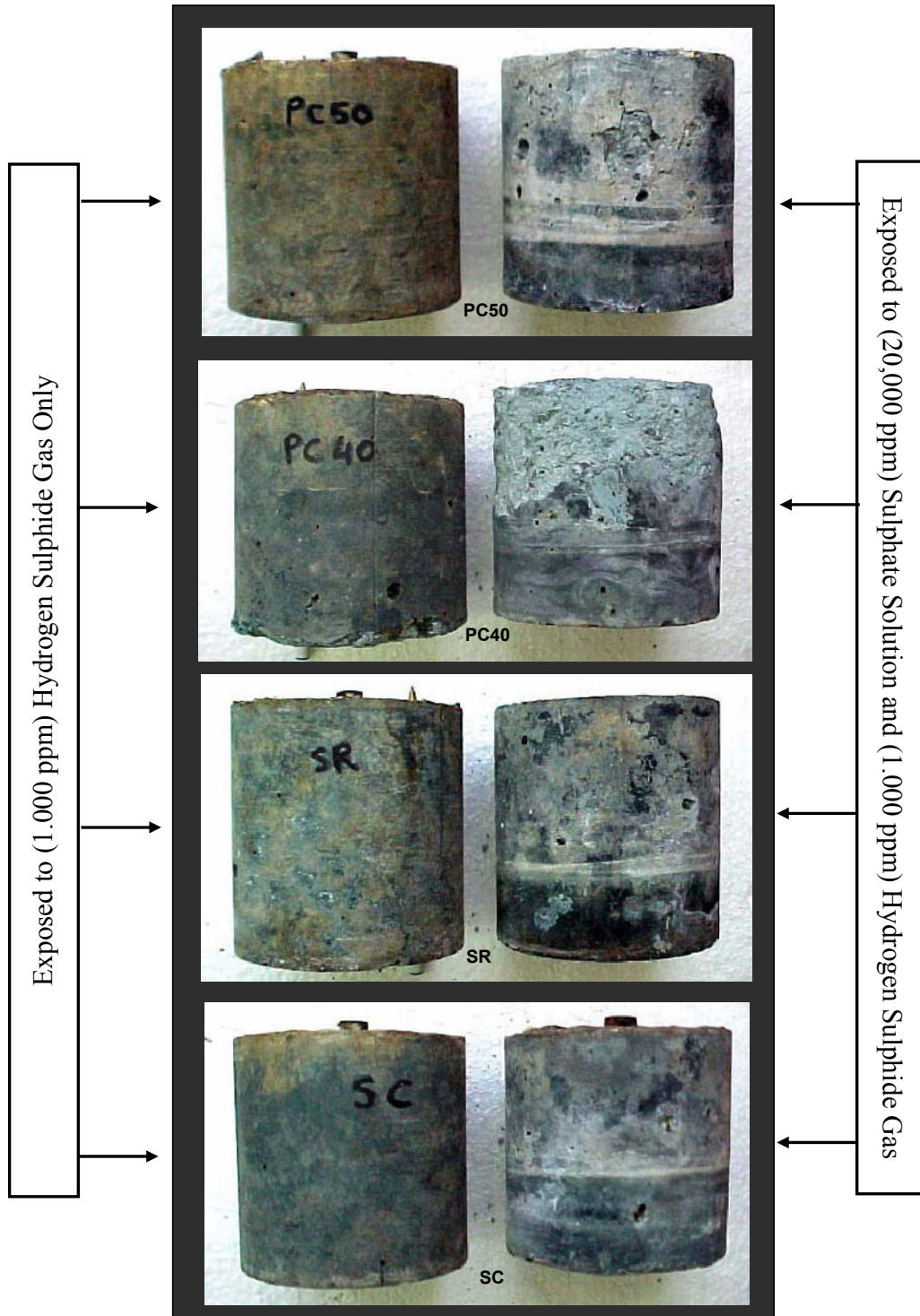
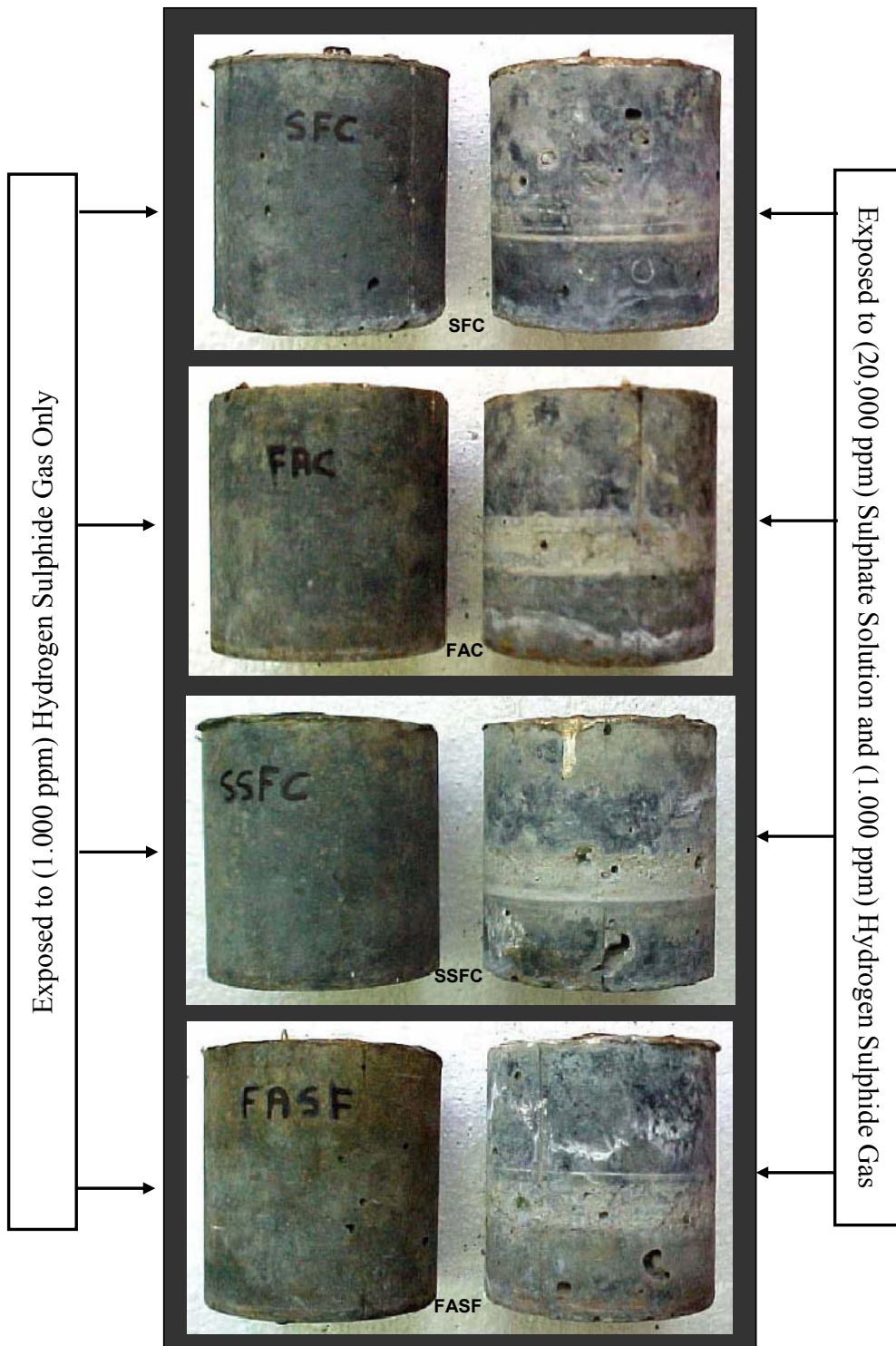


Fig. 6a. Pictures of specimens after the 23<sup>rd</sup> cycle showing the concrete loss.



**Fig. 6b.** Pictures of specimens after the 23<sup>rd</sup> cycle showing the concrete loss.

The results of this study show that, in the sulphide/sulphate environment of manure storage and handling structures made of reinforced concrete, concrete corrosion is the critical factor, steel corrosion is less of a problem. The results indicate that the quality of concrete is the key to the best protection for the steel against sulphate and sulphite attack. It is relatively simple to produce such concrete by making an impermeable, well-cured concrete using a reasonably low W/CM ratio. However, the results also indicate that high Portland cement content creates a problem. Therefore, partial replacement of the Portland cement or the use of sulphate resistant cement will benefit the durability of the concrete of manure structures against the sulphates present in manure.

In summary, it is recommended that reinforced concrete in areas in a livestock building exposed to corrosive materials from manure should be specified to have:

1. high quality, with a maximum W/CM ratio of 0.45, 0.40 if feasible;
2. a maximum Portland cement content of 350 kg/m<sup>3</sup> in order to limit C<sub>3</sub>A content;
3. 8% of the cementitious material to be silica fume, or 35% to be slag;
4. adequate (50 mm) concrete cover over the reinforcement

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