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THE RATE OF CORROSION OF HARDENED CEMENT PASTES AND MORTARS WITH ADDITIVE OF SILICA FUME IN ACIDS

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ABSTRACT

The rate of corrosion of hardened cement pastes (HCPs) and cement mortars in solutions of nitric acid and acetic acid were compared. Cement mortars were prepared with a different sand-to-cement ratio and fineness of sand. A study on the effect of silica fume on corrosion resistance of cement paste and mortars to acid solutions is also presented. The progress of corrosion was expressed in terms of depth of corrosion related to duration of acid attack. The rate of corrosion of all types of specimens was faster in nitric acid solution than in solution of acetic acid with the same concentration. Cement mortars corroded at a slower rate than HCPs, though the difference was minor. There was also some small difference between the rate of corrosion of different mortars. Corroded layers of cement mortars contained smaller, less observable cracks and shrank less than corroded layers of cement pastes. Addition of silica fume slightly reduced the corrosion rate of both HCP and mortar. The differences in the corrosion rate between various types of specimens were smaller in acetic acid than in nitric acid solutions. The specimens lost weight due to acid attack. Loss of weight per unit volume of cement pastes or mortars increased with the increase of the cement content. © *1997 Elsevier Science Ltd*

Introduction

Hardened paste of Portland cement in concrete is readily attacked by various acids, as are several aggregate types, e.g., limestone. Specimens made of plain cement paste are often used to study the corrosion resistance of a cementitious material. The exposure of the hardened cement paste (HCP) specimens to various acid solutions brings out a rapid deterioration of the material's external surfaces. Acids react with hydrated and unhydrated compounds of hardened cement paste and decompose them. The end products of the chemical reactions are generally the calcium salts of the acid and a decalcified residue of the cement hydration products. In the case where the calcium salt of the acid is well solubilized in water, it is

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removed by leaching into the aggressive solution. The mass of the less soluble corrosion products remains on the surface of the specimen, where it forms a porous decalcified layer (or zone) that holds approximately the original shape of the specimen. This layer is composed predominantly of amorphous hydrated oxides: gels of SiO_2.aq., Al_2O_3.aq., and Fe_2O_3.aq., with a small residual amount of CaO or MgO (1–12). However, at pH less than about 3 to 4, it also dissolves Al_2O_3.aq., and at pH less than 1.5 to 2, it dissolves Fe_2O_3.aq. (1–3,11). The porosity of the layer of the corrosive products is extremely high; Bajza et al. (7–10) reported values about 80% (completely corroded HCPs with a water-to-cement ratio (w/c) of 0.4 in 3% solutions of acetic and formic acids) and larger pores than in the unaffected part of the specimen. The most aggressive are strong (fully ionized) acids and those that produce highly water-soluble calcium salts, such as nitric and hydrochloric acids (1–3). Acids giving less water-soluble calcium salts form less porous and less permeable layers of corrosive products when the calcium salts remain in the mixture of products of corrosion. This article presents data on corrosion of HCPs and cement mortars in solutions of nitric (strong) acid and acetic (weak) acid. Both nitrates and acetates of calcium salts are relatively well solubilized in water.

A layer of the corrosive products exhibits practically no binding properties and usually is clearly distinguishable from the still unaffected or slightly affected part of an HCP underlying it. Transport of the aggressive substance across the porous barrier of the layer of corrosive products to the reaction zone inside the specimen is controlled predominantly by diffusion. Diffusion, being the slowest stadium of the corrosion process, is a factor that limits the corrosion rate (1–3,14,15). The thickness of the layer of corrosive products progressively increases in time and follows a ⩓t function (1–3,7–10,13–19).

There exist a number of factors that control the rate of HCP's corrosion in acid solutions. We will mention the following: 1) the type of attacking acid, 2) the concentration and pH of the aggressive solution, 3) the mobility of the solution, 4) the w/c of the HCP, 5) the type of cement, and 6) mineral admixtures, among others. To assess the extent of corrosion of plain cement paste or the cement-matrix composites (cement mortars or concrete) over long periods of time, mathematical models have been developed to express the extent of corrosion as a function of one or more of the corrosion-controlling factors (1–3,7–10,13–19); for example, depth of corrosion (depth of decalcification) related to duration of acid attack, concentration of the attacking acid, and w/c of the specimen. Because some difference between the rate of corrosion of the plain cement paste and cement-matrix composites is expected, the mathematical equations found to express the extent of corrosion of HCP as a function of time (e.g., 9,10,14–17) cannot be used directly to predict the extent of corrosion of concrete or cement mortars. In general, the diffusional transport of aggressive substance across the corroded layer of the cement-matrix composite should be slower than transport across the corroded layer of plain HCP. According to Rombén (3), the acid will have to go around the aggregate particles ("labyrinth, or tortuosity effect"), and the diffusion path will be lengthened. At the same time, the cross-section will vary locally, thus contributing to a reduction of acid transport and, consequently, the rate of attack. Rombén (3) further cites Krischer (20), "who found experimentally, for some models consisting of a number of balls of equal size laid out in the shape of a grid, that the rate of diffusion (counted on an open cross-sectional area) is reduced by a factor of 1.19–1.9 depending on the grid arrangement used." A considerably larger value was expected if balls of different sizes were used.

Addition of silica fume (SF), fly ash, or other pozzolana have been known to significantly improve the durability performance of concrete and cement mortar in some types of aqueous aggressive environments, for example, sulfates. On the other hand, SF and fly ash are mostly reported to have little or no effect when a cement-matrix composite is under attack by solutions of acids (2,7,10,21–22). It is the principal difference in the corrosion mechanism of various aggressive substances that accounts for this fact.

When, for example, sulfates of K, Na, or Ca attack the cement-matrix composite (expansive attack), the SO_4^{2-} ions diffuse via a liquid phase in the pore system of the cement composite. There they react predominantly with calcium aluminate hydrates and/or calcium hydroxide of the HCP. Due to chemical reactions taking place in the pore system of the HCP, the pH value of the pore fluid either increases or remains unchanged. Calcium silicate hydrates are, therefore, no first-rate subject of direct sulfate attack. Permeability, diffusivity, or tortuosity of the cement-matrix composite is considered to be one of the most important corrosion-controlling factors at otherwise constant conditions. The coarser the pore structure, i.e., the larger proportion of larger (capillary) pores the pore structure contains, the easier the aggressive substances may penetrate into the composite and the faster is the rate of corrosion (e.g., 23). Addition of SF to the cement-matrix composite may play a positive role in reducing its permeability and diffusivity. SF also contributes to a reduction in the portlandite content and an increase in the calcium silicate hydrate content in the cement-matrix composites (23–28). These factors, therefore, have a positive effect on the cement-matrix composite's corrosion resistance.

If an acid (dissolutive) attack is the case, the H^+ ions or molecules of an acid diffuse through a porous layer of the decalcified corrosion products and are consumed by neutralization in a reaction zone of cement-matrix composite. We used the term "reaction zone" for the innermost zone in the layer of corrosion products that is in contact with the mechanically hard body of the uncorroded cement-matrix composite. In this relatively thin zone, most of the decalcification reactions of the cementitious material take place, leaving behind more or less decalcified residue of cement hydration products. This term does not precisely coincide with definitions used by Moskvin et al. (1). It needs to be emphasized that the H^+ ions simply cannot diffuse via the pore fluid in uncorroded HCP. (Cement hydration products are stable at such concentrations of OH^- ions that are higher by orders of magnitude than those in the solution of acid or in the reaction zone inside the cement composite.) For that reason, one cannot expect, we suppose, that the improvements of microstructure, reduction of permeability, and other changes in the uncorroded part of cement-matrix composite (that might be achieved, for example, by adding SF to the mixture) would directly lead to a significant increase in corrosion resistance of the specimen in acids. SF, however, can affect the corrosion resistance of cement-matrix composite indirectly, by changing the diffusion resistance of the corrosion products layer. According to Moskvin et al. (2), the change in the permeability of uncorroded HCP is not sought to have an effect on the process of acid corrosion. Also, in our opinion, it is the diffusion resistance of the layer of corrosion products, and not of the uncorroded part of the specimen, that determines the rate of diffusion (the rate of transfer) of the aggressive substance (H^+) into the reaction zone of the cement-matrix composite. As we have previously mentioned, most authors believe that use of various blended cements, or more specifically of the SF, does not bring any (or any substantial) increase in resistance of cement-matrix composites to acids (2,7,10,21–22). Moskvin (2, p.81), for example, found that Portland cement pastes behaved better than blended cements with pozzolana, or there was no difference between them. In HCl with pH 1, as well as HCl with pH 4, they found no difference between these cements. On the contrary, Nischer (29),

TABLE 1
Grading of Coarse, Medium and Fine Fractions of Natural Silica Sand

Particle size fraction (mm)	Percentage of particle size fraction in the coarse, medium and fine sand		
particle sizes (d_1–d_2)	Coarse sand (C)	Medium sand (M)	Fine sand (F)
2.0–4.0	0.17	—	—
1.6–2.0	4.31	—	
1.0–1.6	54.95	0.48	—
0.5–1.0	40.35	94.38	—
0.315–0.5	0.22	3.14	1.47
0.16–0.315	—	2.00	64.00
0.09–0.16	—	—	29.49
0.045–0.09	—	—	4.56
<0.045	—	—	0.48

for example, claims that acid resistance of high performance concrete with w/c ratios of about 0.35 was decidedly improved by addition of microsilica.

Materials and Methods

Cement

Portland cement was used. Its chemical composition was: CaO 62.42%, MgO 1.37%, SiO_2 19.10%, Al_2O_3 5.87%, Fe_2O_3 3.18%, SO_3 2.49%, insoluble residue 2.13%, and ignition loss 1.85%.

Glass Balls

The laboratory glass balls were of equal size, with a diameter (d) of 2.9 mm and density (ρ) of 2.510 g/cm^3.

Sand

Graded natural silica (quartz) sand was used. The sand was furnished in three fractions denoted as coarse, medium, and fine sand. Accurate grading of the coarse (C), mean (M), and fine (F) fractions of sand was determined by sieve analysis. The gradings of all three sand fractions are given in Table 1. The silica (quartz) sand density was 2.635 g/cm^3.

Silica Fume

Its chemical composition was: CaO 3.30%, MgO 1.70%, SiO_2 about 90%, Al_2O_3 0.80%, Fe_2O_3 1.10%, S^{2-} 0.11%, and ignition loss 1.36%. The specific surface area of the SF was in the range of 15,000 to 25,000 m^2/kg and its density 2.20 g/cm^3.

Acid Solutions

Both nitric acid (strong acid) and acetic acid (weak acid) solutions were prepared with a concentration of 0.2 mol/L, the pH values of the solutions were about 0.7 and 2.8, respectively.

Specimen Preparation and Test Procedure

Two separate sets of experiments were performed. In the first set of experiments, a plain cement paste and cement mortars with no addition of SF were prepared; in the second, the pastes and cement mortars with addition of SF were prepared and tested. In this article, the term "cement mortar" will also refer to cement-matrix composite containing glass balls.

Experiments with Specimens Containing No Addition of Silica Fume

Three types of test specimens were prepared: 1) specimens of plain cement paste (control specimens), 2) specimens of cement composites containing glass balls (glass ball composites), and 3) specimens of cement mortars containing silica sand. The w/c values of the control specimens and the cement composites with balls were 0.3, 0.4, and 0.5. Mortars were prepared only with a w/c of 0.5. The mixtures' proportions are given in Table 2. The test specimens were prepared using glass tubes filled with the cement paste, glass ball composites, or cement mortars. Three specimens with the same composition were prepared from each batch. The specimens were consolidated by slight tapping. The inner diameters of the tubes were 3.2 cm or 1.4 cm; the tubes were open on one side only. The specimens were cured for 24 h at 20°C and 100% room humidity, then in lime water for another 27 days, and finally they were placed into aggressive solutions of nitric or acetic acid. The concentration of each solution was kept at a constant value ($c = 0.2$ mol/L). All specimens in the aggressive solutions were hung on a string, with the reaction surface facing downward (16). Depth of corrosion of the specimens was measured regularly during the experiment with calipers. The depth of corrosion was defined as the distance between the end of a glass tube (the surface of the specimen before corrosion) and the boundary between the corroded and uncorroded specimen that was distinguishable visually.

Experiments with Specimens Containing Addition of Silica Fume

Test specimens of cement pastes containing 0%, 5%, 10%, and 30% of SF by weight of cement, and cement mortars containing 0% and 10% of SF by weight of cement were prepared (Table 3). Water-to-cement ratio of the specimens was 0.5. The test specimens were of the same type as those mentioned above. The method of preparation and consolidation of

TABLE 2
Proportions of Components in Mixtures Containing No Addition of Silica Fume

Batch No.	Designation	Sand/ Cement*	Weight proportions of components Glass balls	Sand fraction C	M	F	Cement	W/C
1	A	—	—	—	—	—	1	0.5
2	B	1	1	—	—	—	1	0.5
3	C	3	3	—	—	—	1	0.5
4	D	1	—	⅓	⅓	⅓	1	0.5
5	E	3	—	1	1	1	1	0.5
6	G	2	—	1	—	1	1	0.5
7	H	3	—	3	—	—	1	0.5
8	J	1	—	—	—	1	1	0.5
9	A2	—	—	—	—	—	1	0.4
10	B2	1	1	—	—	—	1	0.4
11	A3	—	—	—	—	—	1	0.3
12	B3	1	1	—	—	—	1	0.3

* Sand/Cement is the sand-to-cement ratio by mass. It also includes the glass balls-to-cement ratio.

the test specimens, the arrangement of a corrosion test and conditions of tests were identical with that in the experiments described above. The exception was 60 days-long period of hardening of the specimens in the lime water and different orientation of the test specimen in the aggressive solutions (test specimens were oriented vertically with the reaction surface oriented upwards). In this set of experiments there was also measured shrinkage of the corroded layers and mass loss referred to a constant reaction area. The shrinkage of the corroded layer was defined as a difference between the depth of corrosion and the thickness

TABLE 3
Proportions of Components in Mixtures Containing Addition of Silica Fume

Batch No.	Designation	(S + SF) / Cement	Weight proportions of components SF	Sand fraction C	M	F	Cement	W/C
21	P	—	—	—	—	—	1	0.5
22	P-SF-5	0.05	0.05	—	—	—	1	0.5
23	P-SF-10	0.10	0.10	—	—	—	1	0.5
24	P-SF-30	0.30	0.30	—	—	—	1	0.5
25	MC	3	—	3	—	—	1	0.5
26	MC-SF-10	3	0.10	2.9	—	—	1	0.5
27	M	3	—	1	1	1	1	0.5
28	H-SF-10	3	0.10	0.9666	0.9666	0.9666	1	0.5

Abbreviations: S, sand; SF, silica fume; P, paste; MC, mortar with coarse sand; M, cement mortar.
*Sand/Cement is the sand-to-cement ratio by mass. It also includes the glass balls-to-cement ratio.

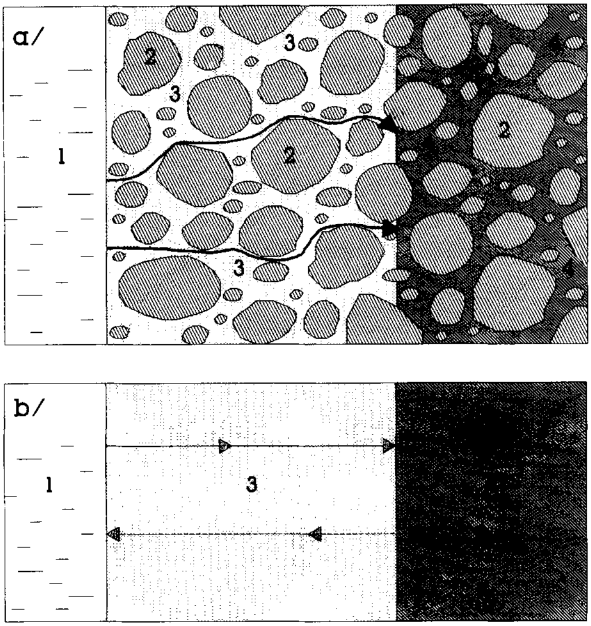

FIG. 1.
Corroded specimens after 30 days in nitric acid: (*a*) control specimen of plain cement paste (Batch 1), (*b*) cement matrix composite containing glass balls (Batch 2), (*c*) cement mortar from fine sand (Batch 8).

of corroded layer (or as a distance between the end of a glass tube and the external surface of the corroded specimen). It was measured using calipers.

Results and Discussion

Experiments with Specimens Containing No Addition of Silica Fume

Figure 1 shows the corroded specimens' appearance after 30 days of exposure in a solution of nitric acid. The corroded (decomposed) cementitious material was white. The narrow dark (orange-brown) ring in the corroded layers represents a zone with an increased content of hydrated ferric oxide (1,3,4,6,11). Figure 1a shows long cracks running across the corroded layer of the control specimen of plain cement paste (Batch 1). Such cracks make it possible to transport the dissolved matter besides diffusion, also by convection, and thus increase the transport rate. The cracks in the corroded layer of the specimen with glass balls (Batch 2) spread radially around the glass balls (Fig. 1b). The cracks developed as a result of volume contraction of the cementitious material in the process of its decalcification and conversion into a mass of corrosive products. The corroded layer in the cement mortar with fine sand (fine sand: cement = 1:1, w/c = 0.5) shown in Figure 1c does not contain any visible cracks. It is uncertain whether any smaller, radially spreading cracks exist around small particles of sand. The consequence of the volume contraction of corroded HCP is shrinkage of corroded layers of specimens (reduction of the corroded layers' thickness). The cracks' formation and

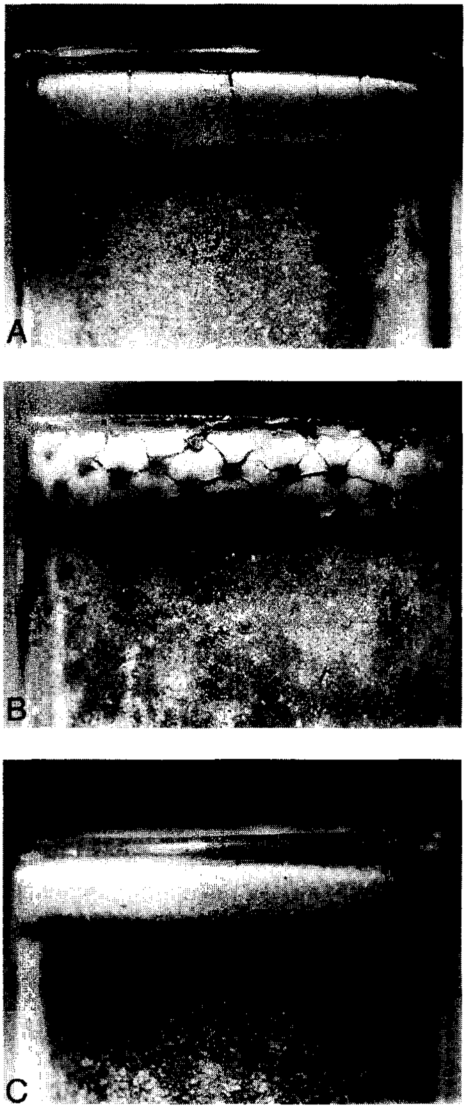

FIG. 2.
Corrosion of control cement paste specimens and specimens containing the glass balls with various w/c in the solution of nitric acid.

shrinkage of the corrosion products layer of plain cement paste is, therefore, considerably higher (see Fig. 1a and first line in Table 4) than those of cement mortars. Cement mortars showed no measurable values of shrinkage (by the method used).

The glass balls incorporated in the cement paste only slightly reduced the rate of corrosion of the specimen (Figs. 2 and 3). The influence of glass balls did not depend on the w/c of the specimens.

The progress of corrosion of cement paste and cement-matrix composites (mortars and

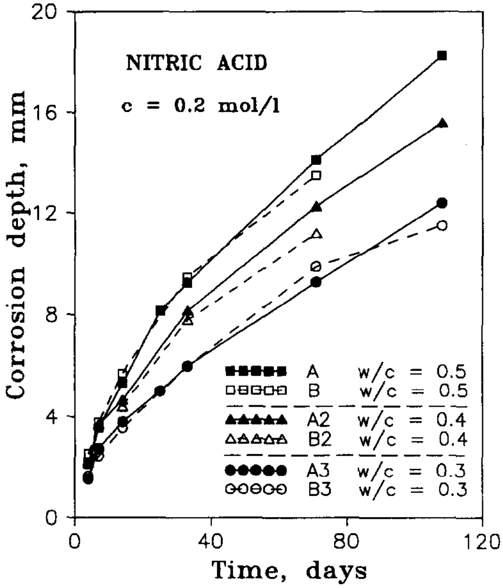

FIG. 3.

Corrosion of control cement paste specimens and specimens containing the glass balls with various w/c in the solution of acetic acid.

glass ball composites), with a w/c of 0.5, which were subjected to attack by nitric acid and acetic acid solutions, relatitve to time, are shown in Figure 4 and Figure 5, respectively.

All cement-matrix composites corroded in nitric acid solution more slowly than the control specimen of plain cement paste (Fig. 4). There are at least three possible explanations for this fact: 1) there are smaller and less numerous cracks in the corroded layers of cement-matrix composites (especially of mortars) than in the corroded layers of plain cement paste, and the consequence is slower transport of matter through the corroded layers of cement matrix composites; 2) there is very small or no measurable shrinkage (i.e., the thickness reduction) of corroded layers of cement-matrix composites; and 3) there is greater tortuosity (labyrinth effect) in the corroded layer of cement matrix composites than in corroded layers of cement pastes. All these factors contribute to the increase of the diffusion resistance of corroded layers of cement-matrix composites.

Incorporation of sand reduced the corrosion rate of the cement-matrix composites more effectively than incorporation of glass balls. It is obvious that smaller, angular and evenly dispersed sand particles prevented formation of large cracks more effectively than glass balls (Figs. 1c and 4).

The rate of corrosion of cement paste and cement-matrix composites was generally slower in acetic acid than in nitric acid solution (Fig. 5). The difference between the corrosion rate of various cement-matrix composites and the hardened cement paste in acetic acid solution was also relatively very small. The attack of acetic acid on the specimen of cement paste led to formation of a light brown corrosion products layer. This layer contained only small cracks on its surface, and shrank less, than the corroded layer of cement paste that had been formed in nitric acid solution (first line in Table 4). Accordingly, from the three hypothetical factors that have been considered to reduce the corrosion rate of cement-matrix composites in

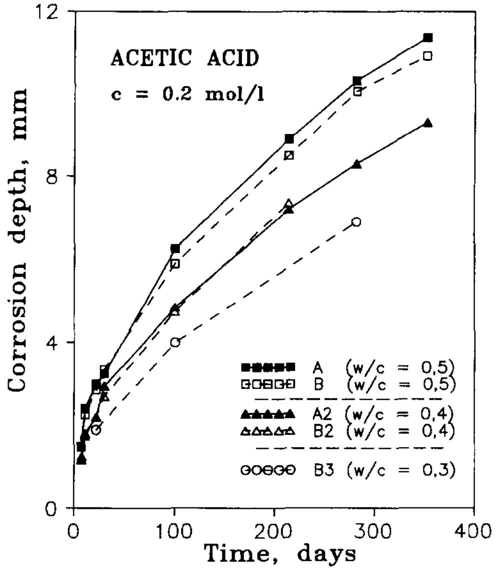

FIG. 4.
Corrosion of the specimens with w/c = 0.5 in an aggressive solution of nitric acid.

comparison to cement pastes in nitric acid solution, we consider the first two to be less effective at acetic acid attack. There might also exist some unspecified difference between corrosion mechanisms in nitric and acetic acids, that could also affect the corrosion rates.

The fastest rate of corrosion in acetic acid solution was found, surprisingly, in specimen

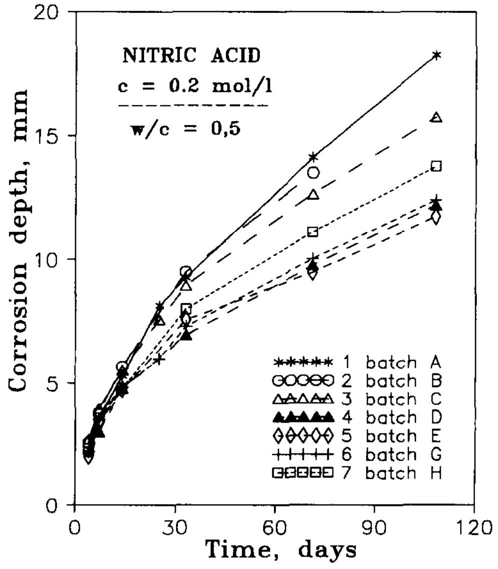

FIG. 5.
Corrosion of the specimens with w/c = 0.5 in an aggressive solution of acetic acid.

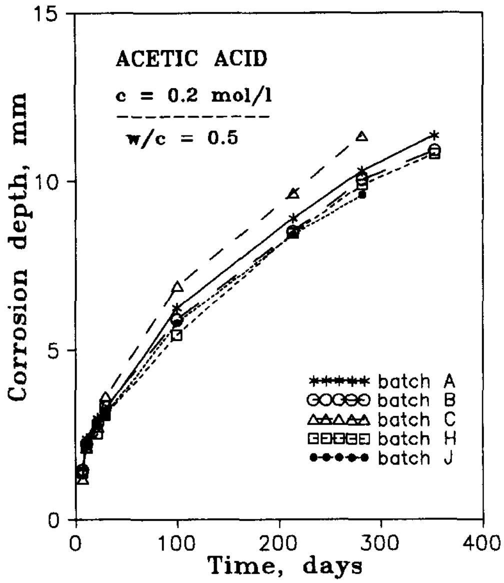

FIG. 6.
Corrosion of the specimens with w/c of 0.5 in an aggressive solution of nitric acid.

3, which contained glass balls (Batch C). It was probably the result of experimental error, caused probably by unhomogeneity on the surface of these specimens. (During preparation of the test specimens the glass balls incorporated in the fresh cement paste were settling down. This had probably driven out some cement paste with a lower w/c ratio to the surface of the specimens.)

Experiments with Specimens Containing Addition of Silica Fume

Results of experiments on corrosion of test specimens in nitric acid solution confirmed our knowledge of the slower rate of corrosion of cement mortars compared with pastes (Fig. 6). Mortars containing all three fractions of sand (with better grading) behaved better than those containing only coarse sand.

The addition of SF to cement pastes slightly improved their resistance to nitric acid attack. The effect of adding 5% and 10% SF to cement pastes was very small; the effect of a 30% addition of SF was considerably higher (Fig. 6). The addition of SF also reduced shrinkage of layers of corrosion products and formation of cracks in them. The cracks in the layer of corrosion products of cement paste containing 30% SF were finer, smaller, and less numerous than the cracks in the layer of corrosion products of plain cement paste and cement pastes containing 5% or 10% SF. Measurement of shrinkage of corroded layers supported these observations (Table 4). The values of shrinkage decreased with the SF content in the cement paste. SF added to cement pastes, besides having reduced the shrinkage of the corroded layers and the formation of cracks in them, also probably had some intrinsic effect on diffusion resistance of the corroded layers.

TABLE 4
Shrinkage Per Length of the Layers of Corrosive Products of Cement Pastes with w/c = 0.5 Exposed for about 300 Days to Solutions of Acids

Batch No.	Designation	Nitric acid solution		Acetic acid solution	
		shrinkage (mm)	shrinkage (%)	shrinkage (mm)	shrinkage (%)
21	P	3.15	12.8	0.70	7.0
22	P-SF-5	2.90	12.2	0.63	5.6
23	P-SF-10	2.68	11.8	0.50	4.9
24	P-SF-30	1.63	8.6	0.30	3.2

Corroded layers of cement mortars had no measurable shrinkage and contained no visually observable cracks.

Experimental results on corrosion of cement pastes and mortars containing additional SF in acetic acid solution are presented in Figure 7 and Table 4. The rate of corrosion of the specimen in acetic acid was, in general, lower than in the nitric acid solution. The difference between corrosion rates of various types of specimens in acetic acid was relatively very small. The addition of SF did not change the corrosion rate noticeably. Corroded layers of cement pastes that had developed in acetic acid contained only tiny cracks, and only on the surface of the corroded layer; the layers shrank less than the corresponding ones from nitric acid.

The specimens subjected to attack of acetic and nitric acids were lower in weight. Loss of weight of the specimens related to depth of corrosion are shown in Figures 8 through 10. The relationships are linear; they are expressed by best fitting lines. Each line characterizes decomposition of cementitious material in a specific type of specimen. Loss of weight (in grams per square centimeter) corresponding to a corrosion depth of 10 mm represents also a loss of weight per unit volume of the specimen (in grams per cubic centimeter). The value is obviously proportional to cement content in the unit volume of the specimen. We might expect some small deviations in this proportionality, depending on the cement type used in

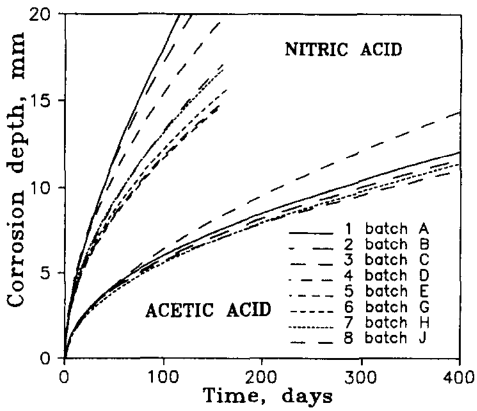

FIG. 7.
Corrosion of the specimens with w/c of 0.5 in an aggressive solution of acetic acid.

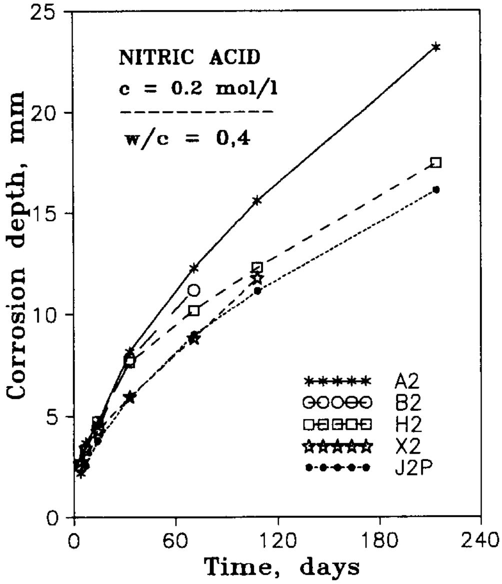

FIG. 8.

Loss of weight related to depth of corrosion of specimens subjected to nitric acid attack.

various types of specimens (if plain Portland cement or blended cement with SF was used). This is because there exists a difference in hydration product content (e.g., in calcium hydroxide content) between these two cements.

Weight loss values of specimens subjected to the action of nitric acid were slightly higher

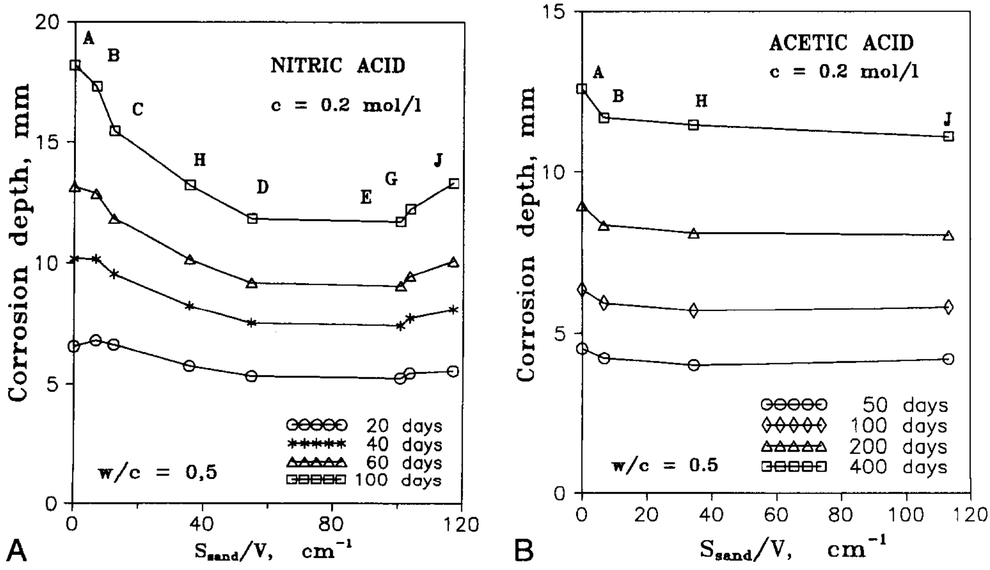

FIG. 9.

Loss of weight related to depth of corrosion of specimens subjected to acetic acid attack.

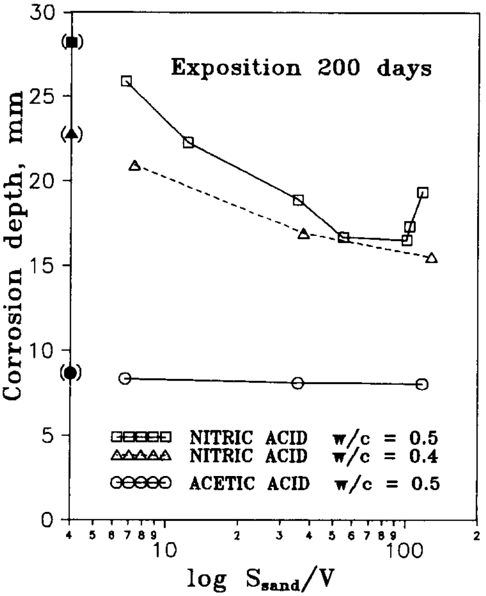

FIG. 10.

than those of specimens subjected to attack by acetic acid. This, we suppose, is prevailingly the result of deeper decomposition of the cementitious material in nitric acid (16).

Conclusions

The rate of corrosion of cement pastes and cement mortars is higher in nitric than in acetic acid solutions.

The rate of acid corrosion of cement mortars is slower than that of cement pastes. Sand particles prevent formation of long cracks in the layer of corrosion products, reduce the number of cracks in a unit volume of the corroded layer, and reduce shrinkage of this layer. Sand particles furthermore increase the diffusion pathway of the aggressive agent in the corroded layer due to a tortuosity effect. The difference between the corrosion rate of cement mortars and of cement pastes in nitric acid is noticeable. The difference is negligible if acetic acid attack occurs.

Addition of SF to cement paste or cement mortar slightly reduces their corrosion rate in a solution of nitric acid. The positive effect of SF is, however, of little significance, in our opinion. Addition of SF does not change the corrosion rate noticeably in a solution of acetic acid.

Loss of weight of cement pastes and cement mortars being attacked by nitric and acetic acids is proportional to cement content in the unit volume of the specimen.

The quantity of an acid that is expected to be consumed by chemical reactions with the cementitious material contained in a unit volume of specimen will be proportional to the cement content in the given specimen. A given quantity of an acid, therefore, will decompose just such volume of the cement paste or mortar, where cement content is proportional to acid quantity. Or a given quantity of an acid will cause the deeper corrosion of a cement mortar the less cement it contains.

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