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Resistance of Mortars Containing Silica Fume to Attack by a Solution Containing Chlorides

by R. F. Feldman and Huang Cheng-yi

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RÉSUMÉ

Des mortiers ayant des rapports eau/ciment + poudre de silice (e/c+s) de 0,45 et 0,60 ont été exposés à une solution contenant un mélange de chlorures de Mg, de Ca et de Na afin d'évaluer leur résistance à l'attaque. Les mélanges contenaient 0, 10 et 30% de poudre de silice, et le rapport sable/liant était de 2,25. La consistance, la distribution du diamètre des pores, la teneur en Ca(OH)_2 et en eau non évaporable ont été mesurées avant et après exposition à la solution de sels. L'inclusion de poudre de silice s'est traduite par une augmentation de la durabilité du mortier, notamment dans des spécimens ayant un rapport e/c+s de 0,45 et durcis pendant 28 jours. Des spécimens de même rapport titrant 30% de poudre de silice se sont aussi révélés durables, même après sept jours de durcissement. L'hydroxyde de calcium a été éliminé des mortiers à la suite de l'exposition à la solution de sels.

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RESISTANCE OF MORTARS CONTAINING SILICA FUME
TO ATTACK BY A SOLUTION CONTAINING CHLORIDES

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ABSTRACT

Mortars prepared at water/cement + silica fume ratios, $w/(c+sf)$, of 0.45 and 0.60 were exposed to a solution containing a mixture of Mg^- , Ca^- and Na^- chlorides to investigate their resistance to attack. Mixes contained 0, 10 and 30% silica fume, and sand-binder ratio was 2.25. Stiffness, pore-size distribution, $Ca(OH)_2$ content and non-evaporable water were measured before and after exposure to salt solution. Inclusion of silica fume resulted in increased durability of mortar, especially in specimens prepared at $w/(c+sf)$ of 0.45 and cured for 28 days. Specimens prepared at the same $w/(c+sf)$ with 30% silica fume were also durable even after seven days of curing. Calcium hydroxide in mortars was reduced to zero by exposure to the salt solution.

Introduction

Corrosion of concrete by salt solutions has been studied for many years (1), most often using sulphates, sea water and magnesium chloride (2-6). Although much attention has been directed to anions such as SO_4^{--} , it is recognized that cations are always involved in the deterioration process. Solutions containing Mg^{++} are particularly aggressive. Mg^{++} may react with $Ca(OH)_2$ to form $Mg(OH)_2$, and in the presence of Cl^- may form magnesium oxychloride ($Mg_2(OH)_3Cl \cdot 4H_2O$). Calcium silicate hydrate may also be gradually broken down (2).

The solubility of many of these corrosion products is enhanced in the presence of sodium and calcium chlorides (5), and this adds to the complexity of the mechanism of attack by a mixed solution. A major factor is the leaching out of reaction products that results in softening of the specimen. The most effective action against such forms of attack is to use concrete with the reduced permeability obtained by adding blast furnace slag and fly-ash blends (7-10). Prolonged curing is always needed, however, and this poses practical problems. Because silica fume is more reactive than fly-ash or slags, it is expected that durable concretes can be obtained with shorter curing times by using it as the additive (11-13).

The object of the present study was to determine the effect of mixed chloride solutions on mortar discs containing silica fume and to relate these properties to pore structure, calcium hydroxide, and non-evaporable water contents.

Experiment

Materials

Type I cement with a C_3A content of 11.82% and silica fume containing 95.2% SiO_2 , 1.56% carbon, 0.27% K_2O , 0.10% Na_2O (surface area 21 000 m^2/kg) were used. Ottawa silica sand conforming to ASTM-C109 was used for mortar with a sand-binder ratio of 2.25. Cement in the mortar contained either 0, 10 or 30% silica fume. Mixes were prepared at $w/(c+sf)$ ratios of 0.45 and 0.60. Cylinders 75 × 160 mm were made from six different mixes; they were sliced into discs 75 mm in diameter and 6.4 mm thick after curing for 24 h at 21°C. Curing was carried out in saturated lime water at 21°C for seven and 28 days. Specimens were exposed to 4% $MgCl_2$ solution for 150–170 days, followed by exposure to a solution of 27.5% $CaCl_2$, 3.9% $MgCl_2$, 1.2% $NaCl$, and 2.1% $NaHCO_3$ (14). The solution was renewed approximately every thirty days.

All specimens exposed to the salt solution will be referred to according to the following scheme:

Silica fume, %	$w/(c+sf)$	
	0.45	0.60
0	C^L	C^H
10	B_{10}^L	B_{10}^H
30	B_{30}^L	B_{30}^H

Properties

Load-deflection measurements were carried out in flexure; discs were loaded at a deflection rate of 0.5 mm/min to a maximum load of 8 kg; seven discs were used to obtain the value for each specimen. Measurements were made as a function of time of exposure to the salt solution and the results following exposure to $Ca(OH)_2$ solution were used as reference. Deflection was measured on a saturated mortar disc loaded at its center and supported at three points on the circumference (14). $Ca(OH)_2$ and non-evaporable water contents were determined as follows: TGA of mortars, by a Cahn Balance using a 1000-mg sample; weight loss between approximately 400 and 600°C and 100 and 1000°C by determining $Ca(OH)_2$ and non-evaporable water contents, respectively. These values were obtained before and after exposure to the salt solution. Pore size distribution was determined as follows: mercury intrusion of specimens was performed up to a pressure of 408 MPa before exposure, after seven and after 28 days of curing, and after various periods of exposure to salt solution. Samples were dried by vacuum and final heating for 8–14 h at 100°C prior to taking these measurements.

Results

Reflection measurements

The time dependence of deflections in flexure are presented in Fig. 1(a-c). The reference specimens in Fig. 1(a) were measured after 28 days of initial curing and show a decrease in deflection for a further 15 days; after this values remain relatively constant although there is some variation between 100 and 250 days, possibly owing to the measurement technique.

MORTARS, SiO_2 FUME, RESISTANCE, CHLORIDE, ATTACK

FIG. 1
Deflection of mortar specimens in flexure during exposure to salt solution.
(a) Reference specimens in $\text{Ca}(\text{OH})_2$ solution
(b) 7-day cured specimens

MORTARS, SiO_2 FUME, RESISTANCE, CHLORIDE, ATTACK

The results for seven-day cured specimens presented in Fig. 1(b) also show decreases in deflection in the first ten days. Specimen B_{30}^L shows a small gradual increase over 325 days of exposure, while B_{10}^L and C^L (which behave similarly) differ from B_{30}^L after about 180 days in that deflections increase markedly. The specimens prepared at the higher $w/(c+sf)$ have, as expected, a higher initial deflection; in B_{10}^H and B_{30}^H deflection increased after about 90 days, in B_{30}^H more rapidly, but after 313 days both specimens failed, with deflections of 0.0725 and 0.0925 mm for B_{10}^H and B_{30}^H , respectively. Deflection of C^H started to increase after 170 days and attained 0.0650 mm after 420 days. At higher $w/(c+sf)$ ratios the silica fume addition decreases resistance to salt solution attack because up to seven days the pozzolanic reaction of the silica fume has proceeded only to a limited extent. At low $w/(c+sf)$, however, the silica fume is beneficial, especially for specimen B_{30}^L , which displays a low increase in deflection after 420 days of exposure.

The results for the 28-day cured specimens presented in Fig. 1(c) show no decrease in deflection in the initial period of exposure, as with the seven-day cured and reference samples. Specimen B_{30}^L shows a low rate of increase over 300 days of exposure, as does B_{10}^L . Specimen C^L shows an increase in deflection, especially after 160 days of exposure, but less than the same $C^L(7)$ specimen cured for only seven days. Specimens prepared at a $w/(c+sf)$ of 0.60 are thus much more resistant to salt solution than those cured for only seven days. Specimen $B_{30}^H(28)$ shows less deflection than $B_{10}^H(28)$ and $C^H(28)$, in that order, and after 380 days of exposure shows a value of 0.0435 mm as compared to 0.0925 mm for specimen $B_{30}^H(7)$.

FIG. 1(c)
Deflection of mortar
specimens in flexure
during exposure to
salt solution.
28-day cured
specimens

Pore size distribution

The results for pore size distribution measurements are presented in Figs. 2 to 5 for specimens cured for seven days at $w/(c+sf)$ of 0.4 and 0.6 and for those cured for 28 days at $w/(c+sf)$ of 0.4 and 0.6, respectively. Measurements were made before and after exposure to the salt solution (Table I). The total intruded volume is less for the specimens containing silica fume and exposed to salt solution than for those not so exposed; the greater the silica fume content the less the intruded volume. Specimens without silica fume, $C^L(7)$, $C^L(28)$ and $C^H(28)$, showed slight increase in

FIG. 2

Comparison of pore-size distribution curves of cement mortar before and after corrosion ($w/(c+sf) = 0.45$, 7 days)

(a) $C^L(7)$, (b) $B_{10}^L(7)$, (c) $B_{30}^L(7)$

FIG. 3

Comparison of pore-size distribution curves of cement mortar before and after corrosion ($w/(c+sf) = 0.60$, 7 days)

(a) $C^H(7)$, (b) $B_{10}^H(7)$, (c) $B_{30}^H(7)$

FIG. 4

Comparison of pore-size distribution of cement mortar before and after corrosion
 $(w/(c+sf) = 0.45, 7 \text{ days})$
 (a) $C^L(28)$, (b) $B_{10}^L(28)$, (c) $B_{30}^L(28)$

FIG. 5

Comparison of pore-size distribution of cement mortar before and after corrosion
 $(w/(c+sf) = 0.60, 7 \text{ days})$
 (a) $C^H(28)$, (b) $B_{10}^H(28)$, (c) $B_{30}^H(28)$

MORTARS, SiO_2 FUME, RESISTANCE, CHLORIDE, ATTACK

Results show that Ca(OH)_2 has been totally removed from all the specimens except $\text{C}^{\text{H}}(28)\text{-c}$, where a small amount was unleached.

Specimens $\text{B}_{30}^{\text{H}}(7)$ and $\text{B}_{10}^{\text{H}}(7)$ both failed rapidly, although the former contained only 4.96% Ca(OH)_2 . In both cases, the high $w/(c+sf)$ and the short hydration time ensured their high permeability, and products other than Ca(OH)_2 could be readily attacked or leached.

Specimens $\text{B}_{30}^{\text{L}}(28)$, $\text{B}_{10}^{\text{L}}(28)$ and $\text{B}_{30}^{\text{L}}(7)$ containing silica fume and prepared at low $w/(c+sf)$ proved to be the most durable. These are followed by specimens $\text{C}^{\text{L}}(28)$ and $\text{B}_{30}^{\text{H}}(28)$. It must be concluded that the low permeability provided by the discontinuous structure of blends is the major reason why low w/c and longer curing for $\text{C}^{\text{L}}(28)$ provided a measure of protection.

The pore-size distribution of specimen $\text{B}_{30}^{\text{L}}(28)$ (Fig. 4(c)), which was the most resistant, shows little change over the whole pore-size range, although there is a decrease in porosity of 3.24% and an increase in non-evaporable water of 4.79% as a result of exposure to the salt solution. These values are even greater for the more permeable $\text{B}_{30}^{\text{L}}(7)$ sample with 7.08% for porosity and 10.5% for non-evaporable water. The reduction in porosity does not occur for cement mortars containing no silica fume and is lower at 10% than at 30% silica fume addition. It may contribute to the increased resistance to salt attack with silica fume addition. The increase in non-evaporable water content is greater also at higher silica fume additions. This suggests that reactions occur in the blends that do not occur in mortars containing cement alone. The reactions may involve excess silica fume, low calcium CSH, unhydrated cement and the salt solution; a hydrate is produced.

The action of the salt solution on mortars is clearly the result of several reactions: leaching of Ca(OH)_2 , decomposition of CSH, formation of calcium chloroaluminate, and formation of products involving unreacted silica fume and/or low calcium CSH and Cl^- ions.

Conclusions

1. Silica fume additions, use of low $w/(c+sf)$ ratios, and longer curing periods markedly increase the resistance of mortars to attack by salt solutions.
2. In samples with a small amount of silica fume Ca(OH)_2 content in mortars is greatly reduced by salt solution exposure.
3. Increase in porosity of mortar specimens in the $(97 \div 2) \times 10^3$ nm range as a result of salt solution exposure is followed by reduction in mechanical stiffness.
4. In specimens containing silica fume a reaction occurs with salt solution, silica, unhydrated cement and low calcium CSH that results in reduction of total pore volume and increase in non-evaporable water.

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