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A RAPID TEST FOR SULFATE INGRESS INTO CONCRETE

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ABSTRACT

A rapid test, based on electrochemical polarization, is described for the ingress of sulfate into concrete. The influence of cement type, water/cement ratio and the presence of supplementary cementitious materials on the initial current delivered and total charge accumulated over six hours was investigated. The results correlate with the sulfate penetration depth determined from two year ponding experiments.

Introduction

The degradation of concrete by sulfate attack is complicated. It can be described as a combination of three processes:

- Ingress of the sulfate ion into the concrete pore structure.
- Reaction between the sulfate ion and $\text{Ca}(\text{OH})_2$ and/or $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaSO}_4\cdot 18\text{H}_2\text{O}$ in the cement paste forming the expansive compounds $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ and $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$.
- Expansion and cracking of the concrete with eventual strength loss and disintegration.

The traditional methods of testing the sulfate resistance of concrete (eg. ASTM C452 and C1012) relied on strength changes or expansion of cement paste or mortar immersed in sulfate solution. For ASTM C452, the addition of sulfate to the mix does not represent field conditions and precludes the testing of blended cements. Additionally, both ASTM C452 and C1012 are slow and sensitive to the specimen size and geometry. It was the purpose of this study to develop a rapid, electrochemical test procedure to assess the ability of concrete to resist sulfate penetration.

Experimental

Materials

- *Binder*: Type 10 and 50 Portland cement, silica fume, flyash and blast furnace slag were used for the concrete systems. The chemical compositions are summarized in Table 1.

TABLE 1
Chemical Composition (%) of Cement, Silica Fume, Flyash and Slag

	Type 10	Type 50	Flyash	Silica Fume	Slag
SiO ₂	19.83	20.71	45.2	95.17	35.30
Al ₂ O ₃	4.18	3.77	20.7	0.21	10.62
Fe ₂ O ₃	3.20	4.36	24.83	0.13	0.58
CaO	61.21	62.46	1.63	0.23	36.94
MgO	4.09	3.35	0.97	0.15	13.32
Na ₂ O	0.45	0.35	0.59	0.10	-
K ₂ O	0.89	0.87	2.40	0.27	-
C	-	-	-	1.56	-
L-O-I	1.53	0.88	2.66	2.30	1.16
SO ₃	3.93	2.46	-	0.12	1.41
Free Lime	1.15	0.70	-	-	-

- *Aggregate*: The fine aggregate was an unblended quartz/feldspar sand. A pelitic pemicrite limestone of 19 mm maximum size was the coarse aggregate. All aggregate was tested for alkali reactivity, freeze-thaw durability and petrography.
- *Concrete*: Table 2 summarizes the concrete mix designs. Concrete specimens for ponding were 75 mm x 75 mm x 280 mm prisms. The prisms were coated with wax on five sides for unidirectional sulfate ingress. For the rapid electrochemical sulfate test, the middle 51 mm of 200 mm x 102 mm (O.D.) concrete cylinders was used.

Specimens were ponded in 88 g l⁻¹ Na₂SO₄ solution at 25°C. After two years, the depth of SO₄²⁻ penetration was determined by energy dispersed x-ray analysis (EDXA) following the procedure described previously (1). The base amount of SO₄²⁻ was 2.5±0.5% in all systems. A value of 3% SO₄²⁻ was used to locate the sulfate front.

TABLE 2
Concrete Mix Designs

	Mix 2 (M2) w/c=0.42 (kg m ⁻³)	Mix 3 (M3) w/c=0.5 (kg m ⁻³)	Mix 4 (M4) w/c=0.6 (kg m ⁻³)
Cement	370.0	334.5	279.9
Water	155.4	167.3	168.0
Fine Aggregate	744.6	744.6	762.23
Coarse Aggregate	1117.3	1116.9	1143.35
Slump (mm)	131	143	125
Air (%)	5.9	6.5	4.8

	Cement	Slag	Flyash	Silica Fume	Cure (days)
System 1 (S1)	Type 10	0	0	0	14
System 2 (S2)	Type 50	0	0	10	14
System 3 (S3)	Type 50	65	0	3	28
System 4 (S4)	Type 50	0	30	5	56
System 5 (S5)	Type 50	75	0	3	28

Specimens for the rapid electrochemical sulfate test were prepared in a similar fashion to those for the rapid chloride permeability test (2). The anolyte and catholyte compositions were 88 g l⁻¹ Na₂SO₄ and 12 g l⁻¹ NaOH, respectively. Solutions were replaced hourly, and the test was monitored for six hours. Sixty volts direct current was delivered to the specimen with a Hewlett Packard Model 6274 power supply. Currents were measured with a Hewlett Packard Model 3468B Multimeter.

Results and Discussion

Experimental results are summarized in Table 3. The plots of initial current and total charge versus the sulfate front are given in Figures 1 and 2, respectively. It can be seen that, as the

TABLE 3
Initial Current, Total Charge and Depth of Sulfate Penetration for all Systems

	Initial Current (Amperes)	Total Charge (Coulombs)	Sulfate Front (mm)
S1M2	0.1163	2269	5.0
S1M3	0.2260	3887	7.6
S1M4	0.2940	5738	8.4
S2M3	0.0400	910	2.0
S3M3	0.0370	818	3.8
S4M3	0.0256	575	3.0
S5M3	0.0191	493	2.8

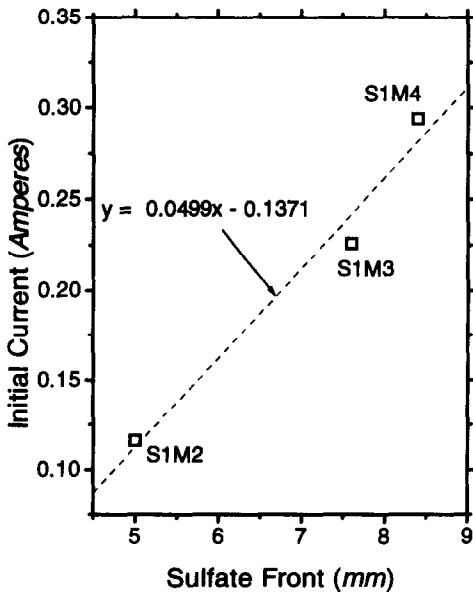


Fig. 1
Initial current versus sulfate penetration depth for System 1 at various w/c ratios.

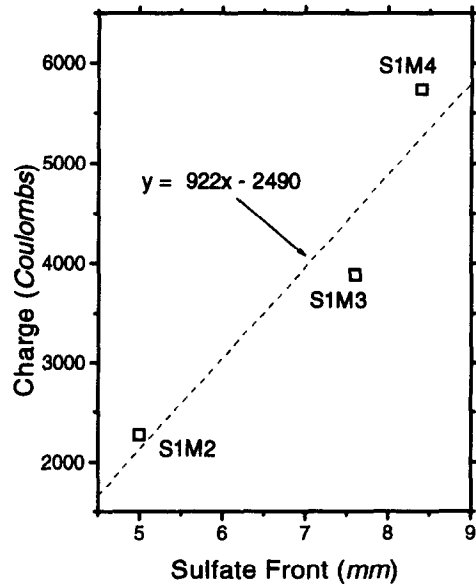


Fig. 2
Total charge versus sulfate penetration depth for System 1 at various w/c ratios.

w/c ratio increases (i.e. S1M2→S1M3→S1M4), there is an increase in the sulfate penetration front because the higher ratios produce a more porous concrete. There is linear relationship between the two year ponded sulfate penetration depth and the initial current and total charge measured with the rapid sulfate electrochemical test.

The influence of the binder composition on sulfate penetration and the relationship to initial current and total charge is presented in Figures 3 and 4, respectively. At constant w/c ratio, the substitution of Type 50 for Type 10 cement results in a significant decrease in the sulfate penetration after two years (i.e. S1M3 cf. S2M3). In addition to a less reactive and expansive paste, the low $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ content in Type 50 cement may produce a less permeable paste. The effect of supplementary cementitious materials on the sulfate penetration and initial current and total charge is also presented in Figures 3 and 4 and in Table 3 (i.e. S2M3, S3M3, S4M3 and S5M3). In all cases, the two year resistance to sulfate penetration, initial current and total charge were low. As indicated in Figures 3 and 4, there is, within a 95% confidence band, a linear relationship between initial current/total charge and sulfate penetration front. Values for S2M3 were not included in the least-squares calculation since values lie at the extreme of the confidence band. This phenomenon for supplementary cementitious materials has been observed previously for the rapid chloride permeability test (3). It has been proposed that the supplementary cementitious material changes the porewater conductivity which will affect current and total charge (4).

Conclusions

1. The preliminary results for the rapid sulfate electrochemical test indicate that the technique is useful for predicting sulfate penetration into concrete.

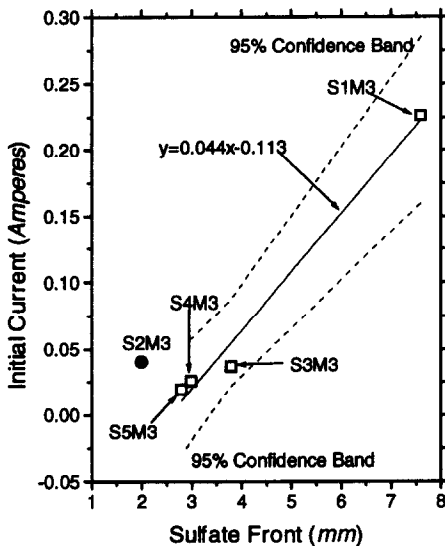


Fig. 3

Influence of cement type and supplementary cementitious materials at constant w/c ratio on initial current and sulfate penetration depth.

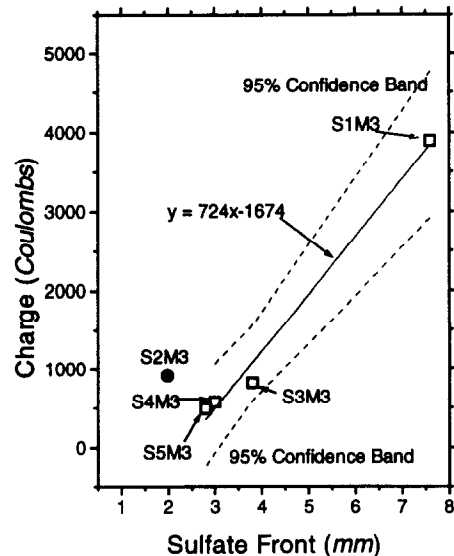


Fig. 4

Influence of cement type and supplementary cementitious materials at constant w/c ratio on total charge and sulfate penetration depth.

2. There is excellent correlation between two year ponded experiments and the initial current or total charge accumulated for concretes with various w/c ratios.
3. The technique corroborates the observation of good sulfate resistance of blended cement concretes based on two year ponding tests.
4. The effects of pore solution composition may be an important consideration especially for blended cement concretes.

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