

Fig. 7--Influence of air entrainment on the permeability of concrete

APPENDIX

The volume of gas passing through a porous medium at a constant rate can be calculated as follows [7]

$$\frac{\mathbf{p} \cdot \mathbf{V}}{\mathbf{t}} = \mathbf{K} \cdot \mathbf{A} \cdot \frac{\mathbf{p}_{c} - \mathbf{p}_{e}}{\mathbf{L}}$$
(1)

or

$$K = \frac{\mathbf{p} \cdot \mathbf{V}}{\mathbf{t}} \cdot \frac{\mathbf{L}}{\mathbf{A}} \cdot \frac{1}{\mathbf{p}_{c} - \mathbf{p}_{e}}$$
(1a)

K = permeability coefficient [m²/sec]where = gas volume $[m^3]$, which flows through the porous medium during time t and at a pressure p V L = thickness of the specimen [m] A = cross-section of the specimen $[m^2]$ $p_{C}-p_{e}$ = pressure difference between top and bottom of the specimen [mbar] t = duration of test [sec]

Eq. (1) is of general validity and does not depend on a special transport mechanism.

The transport mechanism of a gas through a porous media depends on the Knudsen number Kn [7, 8]:

 $Kn = \lambda/r$

with λ = free path of the gas molecules r = capillary radius

For $Kn \ll 1$ stratified flow¹) prevails which can be laminar or turbulent depending on the Reynolds-number. For laminar flow, the permeability coefficient K acc. to eq. (la) can be calculated from the Hagen-Poseuille-law:

$$K = \frac{\mathbf{p} \cdot \mathbf{V}}{\mathbf{t}} \cdot \frac{\mathbf{L}}{\mathbf{A}} \cdot \frac{1}{\mathbf{p}_{c} - \mathbf{p}_{p}} = \frac{r^{2}}{8\eta} \quad \bar{p}$$
(2)

with n = viscosity of the gas

$$\vec{p} = \frac{p_c - p_e}{2}$$
 = mean pressure in the capillary

1) stratified flow means that the impulses between the gas molecules are more significant than their interactions with the surfaces.

Therefore if the transport mechanism is laminar, the permeability coefficient measured at different pressures has to be a linear function of the mean pressure in the capillary. Consequently many authors express the permeability coefficient as:

$$K [m/sec] = \frac{p \cdot V}{t} \cdot \frac{L}{A} \cdot \frac{1}{p_c - p_e} \cdot \bar{p}$$
(2a)

or

$$K[m^{2}] = \frac{p \cdot V}{t} \cdot \frac{L}{A} \cdot \frac{n}{p_{c} - p_{e}} \cdot \bar{p}$$
(2b)

For Kn \gg 1 molecular flow occurs (also Knudsen Flow) which can be calculated from eq. (1a) as follows:

$$K = \frac{\mathbf{p} \cdot \mathbf{V}}{\mathbf{t}} \cdot \frac{\mathbf{L}}{\mathbf{A}} \cdot \frac{1}{\mathbf{p}_{c} - \mathbf{p}_{e}} = \frac{16 \cdot \mathbf{r}}{3} \cdot \frac{1}{\sqrt{2 \cdot \mathbf{R} \cdot \mathbf{T}}}$$
(3)

with R = gas constant

T = temperature

M = molecular weight of the gas

Here, the permeability coefficient is independent of the mean pressure in the capillary.

For concrete with its small pores and especially under low pressures (where the free path of the gas molecules is relative high) a mixture of laminar and molecular flow occurs. Therefore, the test procedure described in this paper has to be evaluated in a general form according to eq. (la). Since the relation between the permeability coefficient and the mean pressure in the capillary is not known, it is important to determine the pressure increase always in the same pressure range (see section "test procedure").

In equation (1a) the gas volume V which flows through the specimen is unknown. Since V or the rate of flow of the gas respectively, are not measured in the test, V has to be calculated from the pressure increase inside the vacuum chamber. From Boyle-Marriotte's law it follows:

$$\mathbf{p} \cdot \mathbf{V} = \mathbf{n} \cdot \mathbf{R} \cdot \mathbf{T} \tag{4}$$

with n = number of molecules

114 Schonlin and Hilsdorf

According to eq. (4) the gas molecules penetrating the concrete specimen occupy a volume V_a at atmospheric pressure ${\tt p}_a\colon$

$$V_{a} = \frac{n \cdot R \cdot T}{p_{a}}$$
(5)

The number of air molecules n can be evaluated from the difference in the number of air molecules in the vacuum chmaber at the beginning and at the end of the experiment.

At the beginning of the experiment the following relation holds:

$$p_{0} \cdot V_{s} = n_{0} \cdot R \cdot T_{0}$$

$$n_{0} = \frac{p_{0} \cdot V_{s}}{R \cdot T_{0}}$$
(6)

and

with V_s = Volume of the vacuum chamber.

At the end of the experiment:

$$p_1 \cdot V_s = n_1 \cdot R \cdot T_1$$

 $n_1 = \frac{p_1 \cdot V_s}{p_1 \cdot V_s}$

and

$$n_{1} = \frac{p_{1} \cdot v_{s}}{R \cdot T_{1}}$$
(7)

)

From equation (6) and (7) we obtain

$$n = n_1 - n_0 = \left(\frac{p_1}{T_1} - \frac{p_0}{T_0}\right) \cdot \frac{V_s}{R}$$
 (8)

Substituting eq. (8) in eq. (5) results in

$$V_{a} = \frac{(\frac{p_{1}}{T_{1}} - \frac{p_{0}}{T_{0}}) \cdot V_{s} \cdot T_{a}}{\frac{p_{a}}{P_{a}}}$$
(9)

From eq. (9) in eq. (1a) we obtain:

$$K = \frac{p \cdot (\frac{p_1}{T_1} - \frac{p_0}{T_0}) \cdot V_s \cdot T_a}{(t_1 - t_0) \cdot p_a \cdot (p_c - p_e)} \cdot \frac{L}{A}$$
(10)

In eq. (10), p is the pressure at which V is determined. According to eq. (5), V is related to the atmospheric pressure p_a . Therefore:

The pressure p_c at one end of the specimen equals p_a :

$$p_{c} = p_{a} \tag{11b}$$

Since the temperature T is constant during one measurement:

$$T_1 = T_0 = T_a \tag{11c}$$

The pressure \mathbf{p}_{e} is the pressure inside the vacuum chamber. This pressure is not constant during an experiment. However it is sufficiantly accurate to estimate \mathbf{p}_{C} from the mean pressure:

$$p_{c} = \frac{p_{1} + p_{o}}{2}$$
 (11d)

Substituting eqs. (11a), (11b), (11c) and (11d) in eq. (10) results in

$$K = \frac{(p_1 - p_0) \cdot V_s}{(t_1 - t_0) \cdot (p_a - \frac{p_1 + p_0}{2})} \cdot \frac{L}{A}$$
(12)

The parameters which are necessary to evaluate the permeability coefficient K from eq. (12) are either known or to be determined in the experiment.

SP 108-7

Laboratory Experience with the Rapid Chloride Permeability Test

by B. Mobasher and T.M. Mitchell

Synopsis: The new rapid chloride permeability test, in which chloride ions are driven into concrete samples electrically over a 6-hour period, is becoming widely used and has been accepted as an American Association of State and Highway Transportation Officials (AASHTO) standard, T277. This paper summarizes the results of an extensive series of laboratory tests with the new method.

Results of an interlaboratory test program provide single operator and multilaboratory coefficients of variation suitable for use in a precision statement in the standard versions of the method. Several possible revisions to the AASHTO standard procedure are examined, but further study is necessary before any can be accepted. Test results on specimens with diameters other than the standard 3.75 in. (95 mm) called for in T277 are found to be easily adjustable to allow comparisons with standard size specimens. Several fundamental properties of concrete, namely water-cement ratio, coarse aggregate type and gradation, and air content, are shown to affect chloride permeability.

<u>Keywords</u>: aggregate gradation; air entrainment; <u>chlorides</u>; coarse aggregates; <u>concretes</u>; <u>permeability</u>; <u>tests</u>; water-cement ratio

118 Mobasher and Mitchell

ACI member Barzin Mobasher is a graduate student in the Department of Civil Engineering at Northwestern University, Evanston, Illinois. His current research is on the ductility of glass fiber reinforced concrete. He did the work presented here while a Graduate Research Fellow at the Federal Highway Administration's (FHWA) Turner-Fairbank Highway Research Center, McLean, Virginia.

ACI member Terry Mitchell is a research materials engineer in the Office of Engineering and Highway Operations Research and Development at the Turner-Fairbank Center. Dr. Mitchell has been with FHWA for 16 years and has been active in developing quality control tests for materials and construction and in improving quality assurance specifications.

INTRODUCTION

Concrete's ability to delay the initiation of corrosion of its reinforcing steel depends on the concrete's impermeability to chloride ions introduced in deicing salts. Until recently, highway departments and concrete producers have had to rely on ponding tests to assess chloride permeability. Typically in tests such as AASHTO standard test T259 (Ref. 1), small concrete slabs are ponded with 3 percent sodium chloride solution for 90 days; samples are then removed from the slabs and pulverized, and the chloride contents of various depth increments are established by laboratory titration procedures.

In 1983, a new AASHTO standard, T277, was adopted, in which chloride ions are driven into concrete samples electrically over a 6-hour period. The total electrical charge passed during that period has been shown (2) to correlate well with the chloride ion profiles found in various concretes after the 90 day ponding test. Because of its speed and good within-laboratory repeatability, the new test has become widely used by State highway agencies and by manufacturers of specialized concretes. Acceptance of the procedure as an American Society for Testing and Materials (ASTM) standard is also being sought currently.

The present paper summarizes the results of an extensive series of tests with the new method. The goals of the studies included development of: (1) a proposed precision statement for the method; (2) conclusions about the importance of several factors in the method such as variations in the sample conditioning procedure; and (3) conclusions about the effect of materials and construction variables such as coarse aggregate type and gradation and air content on the chloride permeability of concretes.

EXPERIMENTS

Test Procedures

Except where test parameters were intentionally varied, all chloride permeability tests were conducted according to AASHTO T277, "Rapid Determination of the Chloride Permeability of Concrete."(1) In this procedure 3.75 in. diameter by 2 in. long (95 mm diameter by 51 mm) slices of cores or cast cylinders are the test specimens. The curved side of a specimen is first coated with an epoxy sealant. The specimen is then brought to a standard moisture condition by the following vacuum saturation procedure: Vacuum is applied to the dry specimen for 3 hours and then continued for 1 more hour with the specimen immersed in deaerated water; after that, the specimen is soaked in the same water for an additional 18 \pm 1 hours at atmospheric pressure. The ends of the specimen are then sealed into hollow, polymethylmethacrylate, e.g., Plexiglas, chambers (Figure 1). The chamber containing the top of the sample is filled with a 3 percent sodium chloride solution, the chamber containing the bottom with a 0.3N sodium hydroxide solution. Sixty volts DC is applied across the specimen between copper screen electrodes contained in each chamber. The total charge passed, i.e., the integral of the current with respect to time, during a 6 hour period is a measure of the chloride permeability of the concrete.

Materials and Concrete Mixture Designs

The mix proportions and the fresh concrete properties are shown in Table 1. A Type 1 cement was used in all mixes. Except for mixes L through P, the coarse aggregate was a 19.0 mm (3/4 in.) maximum crushed limestone from Riverton, Virginia. Mixes L and M, part of the study of the effects of different aggregates, employed a crushed granite from Occoquan, Virginia, and a river gravel from Brandywine, Maryland, respectively. Mixes N, O, and P used Riverton limestone as the coarse aggregate but the gradations met the requirements for ASTM sizes 5, 6, and 7, respectively. The fine aggregate was a natural sand from White Marsh, Maryland. Air-entraining agent, Darex AEA, was added to all mixes except the latex-modified concretes. Styrene butadiene latex modifiers were obtained from two different sources. Concretes were mixed in 1 1/2 ft³ (0.042 m³) batches in a pan-type mixer.

Three types of specimens were cast (and mechanically vibrated): slabs 14 x 14 x 4 in. (350 x 350 x 100 mm), cylinders 3.75 in. diameter x 13 in. high(95 mm dia. x 330 mm), and beams 3 x 4 x 16 in. (75 x 100 x 410 mm). Only the slabs and cylinders were used for the tests reported here. All of the portland cement concrete samples were cured under wet burlap for 24 hours, then demolded and stored in a fog room 73 \pm 5 ^oF (23 \pm 3 ^oC). Three cylinders 3.75 in. diameter x 4 in. high (95 mm dia. x 100 mm) were removed from each slab after 14 days in the fog room. All of the cylinders and slab were then returned to the fog room until test

120 Mobasher and Mitchell

time. The latex-modified concrete samples received the same initial 24-hour cure under wet burlap, but were then demolded and air-cured until test time.

RESULTS AND DISCUSSION

Development of Precision Statement

The approach for precision statement development was an interlaboratory test program following ASTM C802, "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials" (3). Full details of the sampling and testing program and the statistical analysis are given elsewhere (4) but are summarized briefly here.

The testing was performed by 11 State, Federal, and private laboratories on specimens from mixes A through D. After 28 days of curing, several of the 3.75 in. diameter x 13 in. long (95 mm diameter x 330 mm) cylinders of each mixture were sawed into 2 in. (51 mm) long specimens. The specimens were then distributed among the laboratories. Each laboratory received three randomly selected, replicate specimens of each of the four concrete mixes. They were instructed to follow the updated version of AASHTO T277 being proposed for ASTM adoption at the time; the updated version does not differ significantly from the AASHTO standard. Laboratories were also asked to conduct their tests within a specific 2-week time period so they would all be testing specimens of essentially the same age.

Table 2 and Figure 2 show the results of the interlaboratory tests. Twelve laboratories had originally agreed to participate in the study, but one, laboratory #7, was forced to withdraw just prior to starting the test program due to an equipment failure. The laboratory numbering system was not changed subsequently, so both Table 2 and Figure 2 lack results for laboratory #7. Table 2 shows the numerical results, the total charge passed in coulombs during the 6 hour test for each replicate specimen, along with the mean, variance, and standard deviation for each group of three replicates. Figure 2 is a plot of the individual laboratory averages. The plots of the averages are very similar for the 11 participating laboratories, thus leading to the conclusion there are no interactions between laboratories and materials.

Examination and rigorous statistical analysis of the data in Table 2 led to exclusion of two data sets from the precision statement development: (1) All of the mixture B results were excluded when the specimens were found statistically to be nonuniform, that is, the permeabilities of specimens from the tops of the cylinders were found to differ significantly from those from the bottoms (possible explanations include coarse aggregate settlement, bleeding, and preferential consolidation). And (2) the concrete C, laboratory 2 data was discarded on

statistical grounds as being too variable, i.e., much more variable than results from any of the other laboratories on the same concrete.

With the outlier data removed, the averages and the within- and between-laboratory standard deviations and coefficients of variation were calculated and are shown in Table 3. The concrete B data is shown for comparison purposes, but it was not used for the precision statement development.

Examining the within- and between-laboratory data for concretes A, C, and D suggested there may be a linear relationship between the standard deviations and the average coulomb values. However, the within- and between-laboratory coefficients of variation each varied only over a 6 percent range, so assuming a constant coefficient of variation and using it in the precision statement appeared to be more appropriate. A conservative approach was warranted, so the largest of three values for each of the two coefficients of variation was used in developing the precision statements.

The proposed precision statements themselves are based on guidelines presented in ASTM C670, "Preparing Precision Statements for Test Methods for Construction Materials" (3), and use the data summarized in Table 3. The recommended statements and associated footnotes, which have been incorporated into the version of the method being considered by ASTM, are:

Single-operator precision: The single operator coefficient of variation of a single test result has been found to be 12.3 percent.^(a) Therefore the results of two properly conducted tests by the same operator on the same material should not differ by more than 35 percent.^(a)

Multilaboratory precision: The multilaboratory coefficient of variation of a single test result has been found to be 18.0 percent. (a) Therefore results of two properly conducted tests in different laboratories on the same material should not differ by more than 51 percent. (a) The averages of three test results in two different laboratories should not differ by more than 29 percent. (b)

(a)These numbers represent, respectively, the (1S%) and (D2S%) limits as described in ASTM Standard Practice C670.

(b)The test method does not require the reporting of more than one test result. The precision statement for the averages of three results is given since laboratories frequently will run multiple samples. The number represents the (D2S%) limit divided by the square root of 3.