



FIG. 1
Decrease in permeability with curing time

Test Samples. Twenty-six-year-old concrete cylinders with mix proportions as shown in Table 1 were sliced using a diamond saw. The top and bottom slices (10 mm thick) were used for the microstructural examination, while the rest of the cylinder was cut into 40 mm thick samples for water permeability testing.

Test procedures

Permeability

Permeability experiments were conducted using the Hearn-Mills permeameter⁹ (Figure 2). The concrete specimens were tested before and after drying. Concrete slices were placed into the permeameter without conditioning. At the end of the test the samples were removed from the permeameter, dried in an oven at 105°C and then vacuum saturated (following the AASHTO T 277 procedure¹⁰). After conditioning the samples were retested for permeability. The permeability tests were continued for 7 days. Porosity was calculated on all the samples by measuring dry, saturated and buoyant weights of the samples.

Microstructure

Specimen preparation. Specimens to be examined using backscattered electron imaging must have smooth, polished surfaces for optimal results. Such surfaces are difficult to achieve with cement paste specimens, and even more so for concrete. Polishing preferentially wears away the relatively soft hydration products, leaving the unhydrated cement and aggregate particles as local high spots on the specimen surface. These will affect the resolution of the images and can also make interpretation difficult. Thus too much polishing can be as bad as too little.

We obtained optimal results by using less polishing than would normally be used for cement pastes¹¹. The specimens were vacuum impregnated with a low-viscosity epoxy and cut using a low-speed diamond saw with alcohol as the coolant/lubricant. The specimens were then dried at 90°C and the cut surface reimpregnated with the same epoxy as before. (This step was critical, as it prevented grit and lead from the polishing lap from filling the pores in the specimen.) The grinding and polishing steps were kept to a minimum:

1. #600 grit, 1 lb. pressure for 1/2 hour
2. 6 micron diamond paste, 2 lb. pressure for 1 hour
3. 1 micron diamond paste on lead lap, 2 lb. pressure for 1 hour

Non-aqueous lubricants were used during all stages of grinding and polishing. The specimens were cleaned ultrasonically in ethanol after each step. They were then sputter coated with carbon for examination in the SEM.

Results and Discussion

The results of the permeability tests for 0.9 w/c ratio samples are presented in Table 2. They clearly indicate the self-sealing phenomenon, especially after oven drying of the samples. Figure 3 shows the permeabilities of four typical samples, from Table 2, before and after oven drying. Prior to drying permeability is constant. After drying, however, permeability is initially several orders of magnitude above that of the virgin specimen, but decreases rapidly with time. The major reduction in permeability occurs in the first 24 hours and remains generally constant after 100 hours of testing. The shape of the continuing hydration and the self-sealing curves is similar; however, the comparison of the rates of reduction in permeability due to self-sealing and due to increased levels of hydration (Figures 1 and 3) indicates that the rates are different. Moreover, the specimens tested in this study were water cured for 26 years (which corresponds to approximately 225,000 hours on the x-axis of Figure 1). At this stage of curing terminal hydration levels have been reached so that the effect of continuing hydration during the course of a permeability experiment lasting 100 hours is negligible. It is possible that on oven drying microcracking exposes previously unexposed clinker, thus increasing rate of hydration to the initial rates, i.e. time 0 in Figure 1.

TABLE 2. Permeability results for 26-year-old concrete of 0.9 w/c ratio, with porosity of 0.19

Sample#	Porosity	Permeability $K \cdot 10^{-12}$ m/s		
		Virgin Specimens, saturated	After oven drying	
			Initial	Final
1	-	0.7	260	20
2	-	1.0	180	20
3	.2	0.4	150	2.5
4	.19	1.0	80	6
5	.18	0.6	140	1.2
6	.2	0.8	110	8.5
7	.19	2.0	128	21