

Migration of Inhibitors in Aqueous Solution Through Concrete

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A study of the migration of corrosion inhibitors in aqueous solution through concrete disks (of ≈ 6 -mm thickness) has been undertaken. Concrete disk specimens were placed between glass vacuum flanges and aqueous solutions of salts and a vacuum applied on one side of the disk caused inhibitors to penetrate the disk. The contact times of the aqueous solution with the disks were varied, and the quantities of solution penetrating the disk were evaluated. The depth of solute migration was measured by surface-sensitive analysis (x-ray photoelectron spectroscopy, XPS) of selected portions of fractured disks. XPS measurements were used to establish whether chemical changes had occurred during solute migration and to determine whether differential migration of solute cation and anion had taken place. The implications of the migration of salts and inhibitors for bridge repair are discussed.

The effectiveness of an inhibitor in bridge decks is influenced by its deliverability to the concrete-rebar interface. The study of a related phenomenon, diffusion of chloride in concrete, has attracted attention as a part of the effort to understand the corrosion of steel reinforcing rods in concrete structures. Investigators have reported on chloride diffusion under various conditions to which concrete is exposed (1-4). Some studies of inhibitor diffusion through concrete have been reported. For example, nitrite ion has been found to diffuse downward from a bonded concrete overlay doped with calcium nitrite (5).

The migration of inhibitors in aqueous solutions is examined using sample test specimens designed to permit accelerated migration of inhibitor solution. The principal objectives of the work are to evaluate the rate of inhibitor migration, to determine whether any change in the chemical nature of the inhibitor occurred as a result of interaction with concrete, and to inquire whether the anion-cation concentration ratio was affected by migration through the concrete disks. If an inhibitor is effective in corrosion screening tests (see Dressman et al., a companion paper in this Record) and is also able to migrate under the present test conditions, then it is a good candidate for larger-scale testing. The use of thin concrete disks permitted acquiring information regarding the migration of inhibitors in a shorter time frame. The combination of short experiment times and sensitive surface analysis methods permitted rapid evaluation of inhibitor migration and determination of the chemical nature of the migrating solutes.

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EXPERIMENTAL

The concrete test specimens were sand-modified concrete disks. Concrete cylinders, 3.0 cm in diameter and approximately 10 cm long, were cast. Portland cement was used and the water-cement ratio was 0.47. The concrete mix contained fine sand at a volume equivalent to that had aggregate been used. The concrete was mixed in a small mixer. A vibration table was used to compact the concrete cylinders. The cylinders were cured in a humidity chamber for 7 days.

The cured concrete cylinders were sliced to form disks approximately 0.6 ± 0.1 cm thick. The edges of the disks were sealed using an epoxy resin, leaving an effective penetration diameter of 1.5 cm. Rubber O-rings were placed on the epoxy surfaces while the epoxy was still tacky to ensure good adhesion between the O-rings and the concrete surface. A schematic representation of the concrete disk specimen is shown in Figure 1. The disks were placed between two glass flanges and connected to a vacuum source.

The concrete disks were pumped on a vacuum line to detect leaks caused by any large voids or defects in the epoxy sealing or in the disks themselves. This pump-out process also provided conditioning and drying of the samples. After pumping on the vacuum line, the disks were attached to a vacuum flask and evacuated using a water aspirator. In this arrangement, one side of the disk was exposed to the vacuum and the other side was exposed to air at atmospheric pressure (see Figure 1). A measured quantity of solution was placed on the air side of the system and a rubber stopper was placed on the open tube to eliminate any solvent loss because of evaporation. Vacuum was maintained on the samples for various periods of time, and the fluid remaining in the reservoir was measured. When surface analyses were to be carried out, the disk was carefully removed from the apparatus, dried at 60°C for 15 to 20 min, and then analyzed using x-ray photoelectron spectroscopy (XPS).

Solutions that were studied as a part of the penetration of inhibitors through concrete were: 0.300 M RbCl; chloride-containing pore solution [composition NaOH (0.300 M) KOH (0.600 M), saturated with $\text{Ca}(\text{OH})_2$] containing 3.5 weight percent (wt.%) NaCl; and chloride-containing pore solutions with inhibitor (0.300 M). In the preparation of the inhibitor solutions, sufficient inhibitor was added to pore solution to achieve a concentration of 0.300 M. The inhibitors of interest in this study were sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) and sodium monofluorophosphate ($\text{Na}_2\text{PO}_3\text{F}$) (MFP).

XPS analyses were performed on selected portions of the specimens using a PHI Perkin-Elmer 5300 electron spectrom-

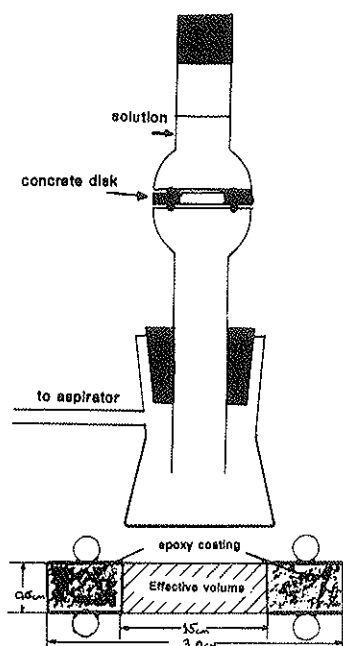


FIGURE 1 Schematic diagram of the solution penetration-aspirator vacuum system. The vacuum could be maintained either with a water aspirator or a conventional vacuum pump. The shaded area of the disk is the region that was coated with epoxy resin. The nominal disk thickness was 0.6 cm. The effective volume exposed to the inhibitor-pore solution is $1.77 \text{ cm}^2 \times 0.6 \text{ cm}$.

eter modified for small-spot measurements (6). Because of the porous nature of the concrete and the amount of water retained in the specimens (even after heating), the samples were maintained at liquid nitrogen temperature (-150°C) for the XPS analysis. The analysis by XPS allows identifying elements that originate from the inhibitor and determining their surface concentrations—thus, the penetration time of solute through the disk. Analysis of the specimens was carried out for upper and lower (vacuum side) portions of fractured disk samples. The spot size for the analysis was $1 \times 3 \text{ mm}$. The results are presented in units of atomic percent (at.%) (6). The binding energy (BE) scale was calibrated by setting the C 1s hydrocarbon (contamination) photopeak binding energy at 285.0 eV (7). The BE data were used to determine the chemical nature of inhibitor elements.

RESULTS AND DISCUSSION

The study of the migration of inhibitor solute species through concrete disks was investigated by (a) determining the volume of solution passing through the disks, and (b) analyzing the upper and lower disk surfaces after exposing the disks to the solution for designated periods of time. The results of the volume transport measurements are shown in Figure 2, in which the volume change is plotted versus the square root of time. In the presentation of the results, the volume of solution

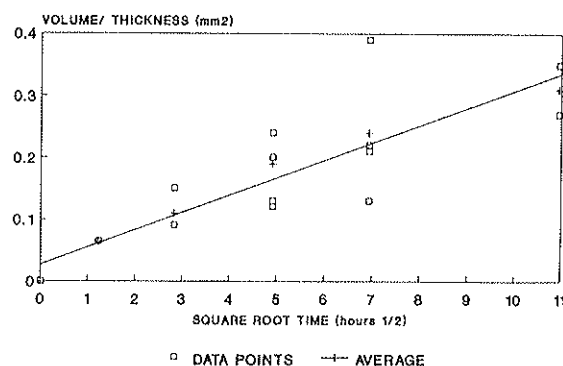


FIGURE 2 Washburn plot of pore solution migration versus square root of time.

migrating into the disks is normalized by dividing the volume transported by the thickness of the disk. In the figure, the data points correspond to the migration of solutions for rubidium chloride, sodium chloride in pore solution, and sodium tetraborate in chloride-containing pore solution. The data points are not distinguished in the figure because the volume change measured for each solution followed the same behavior. The volume change is a linear function of the square root of time. This finding is in agreement with the Washburn equation (8). The significant advantage of the present sample configuration is that the chemical nature of the diffusing species can be determined by subsequent surface analysis of fractured or whole concrete disks.

In the surface characterization measurements, the composition of a concrete (cement + sand) disk was determined to evaluate which elements, at which concentrations, were present on the disk surface, and thus which species could be studied without interference in the migration process. The average elemental composition from measurements on a representative group of disks is presented below. The results are atomic percents.

- C: 20.6,
- O: 56.1,
- Si: 13.9,
- Ca: 6.11,
- S: 1.24,
- K: 1.07,
- Na: .82, and
- Cl: <.2.

The surface chemistry is characterized by silicon, calcium, sodium, potassium, oxygen, sulfur, and carbon. Chlorine was detected at the detection level (<0.2 percent) in some samples. The chemical nature of the metals, inferred from BE measurements, corresponds to that expected for alkali (+1) and alkaline earth (+2) metals. Silicon is present as silicon-oxygen species and sulfur exists as sulfate in these specimens (9). The concentrations of inhibitor elements of interest are low or below the detection limits, and thus detection of inhibitor that had migrated through the disk was possible.

The results for the migration experiments involving aqueous solutions of RbCl are presented in Table 1. Solutions of RbCl were contacted with two disks of different thicknesses, 4.1 and 5.1 mm, for 48 and 120 hr, respectively. The concentrations of rubidium on the top and bottom portions of the disks

TABLE 1 XPS ANALYSIS FOR RbCl PENETRATION THROUGH CONCRETE: SOLUTION PENETRATION RESULTS FOR RbCl (0.300 M)

Disk #	Thickness (mm)	Time (hr)	Surface	C	O	Si	Ca	Cl	Rb
1	4.1	48	Top	45.5	40.0	7.1	5.0	0.3	2.1
			Bottom	32.7	48.4	9.4	6.7	0.5	2.2
2	5.1	120	Top	30.3	49.3	12.1	5.0	0.4	2.1
			Bottom	46.1	37.5	7.2	2.9	0.2	3.8

are approximately equal, indicating that after each time period, the salt had migrated through the disk. Within the experimental error, the chlorine content is the same for top and bottom parts of the disks. However, the chlorine content is significantly less than the rubidium concentration. If equivalent migration had taken place, equal atomic concentrations of rubidium and chlorine should be detected. The migration of rubidium appears, from the present results, to occur more rapidly. To maintain electroneutrality, the migration of rubidium with another anion, as a cation-anion pair, could have taken place, leading to a lower chloride concentration. The identity of another potential anion is not revealed from the present XPS results.

Because the migration experiments involving inhibitors were conducted using chloride-containing pore solution, the migration of pore solution containing 3.5 wt.% NaCl was also investigated. The surface analysis data are presented in Table 2. The analytical results for sodium and chlorine reveal that the cation has migrated through the disk within 8 or 24 hr. The respective sodium concentrations on the top and bottom portions of the two disks are equal. The chloride concentration, either on the top or bottom of the specimen, is less than that for sodium. It appears that an anion exchange process must be taking place with species in the concrete specimen. Elements that could be associated with the cation, including halide ions, were not detected in the XPS measurements. Although sulfate sulfur was detected in the spectra, the concentration of sulfur does not change sufficiently or in a consistent manner such that it could be associated with cation migration. Migration of hydroxide ion along with the cation could take place and provide an explanation for the observed results. An examination of the O 1s photoelectron spectra does not reveal any significant increase in hydroxide oxygen. However, an increase associated with hydroxide migration

TABLE 2 XPS ANALYSIS FOR CHLORIDE-CONTAINING PORE SOLUTION MIGRATION THROUGH CONCRETE: SOLUTION PENETRATION RESULTS FOR PORE SOLUTION + 3.5 wt.% NaCl

Disk #	Thickness (mm)	Time (hr)	Surface	C	O	Si	Ca	Cl	Na
7	5.5	8	Top	49.7	34.1	6.1	2.7	1.9	5.5
			Bottom	43.2	41.4	5.8	5.1	0.4	4.1
8	6.7	24	Top	46.3	35.0	7.2	4.6	2.1	4.8
			Bottom	52.9	34.0	4.9	4.4	0.4	3.3

would be difficult to distinguish because the concentration of oxygen from other chemical species, especially silicon-oxygen- and calcium-oxygen-containing entities, in the concrete is high.

The results for penetration into concrete by sodium borate in chloride-containing pore solution are presented in Table 3. In the table, surface analysis results are presented as a function of time for the upper and lower surfaces of the disks following penetration of the solute. Penetration of borate to the bottom of the disk occurs by at least 24 hr as evidenced by the large boron percent (6.1 at.%) for that sample. This percentage is to be compared with the value of 0.8 at.% boron for the bottom surface of the specimen examined after 1.5 hr. The chlorine and sodium concentrations increase for the 24-hr compared with the 1.5-hr sample. This finding is related to the migration of sodium chloride from the pore solution.

In addition, the silicon and calcium contents are smaller for those surfaces on which inhibitor is present. This result is attributed to the fact that small crystalline particles of inhibitor material were visible on the disk surface, thus blanketing silicon and calcium in concrete. Because the inhibitor solution was in contact with the upper surface of the disks for the duration of these experiments, it is surprising that the boron content for the upper surfaces is not constant. The values range from 5.4 at.% (48 hr) to 1.5 at.% (25 hr). The variation in the percent may be a result of the heterogeneity of concrete and the relatively small spots ($1 \times 3 \text{ mm}^2$) that were analyzed on the disks.

The BE values for the B 1s level in the salt that appeared on the upper and lower disk surfaces, BE = 192.2 and 192.3 eV, respectively, were equivalent to the value, BE = 192.2 eV, measured for 0.300 M sodium borate pore solution frozen on the XPS sample probe. Thus, interaction of aqueous borate with concrete did not result in degradation or decomposition of the inhibitor. These results indicate also that introduction of borate inhibitor to the rebar-concrete interface should be possible.

The experiments with MFP yielded the surface analysis results presented in Table 4. Before considering the results,

TABLE 3 XPS ANALYSIS FOR SODIUM TETRABORATE MIGRATION THROUGH CONCRETE: SOLUTION PENETRATION RESULTS FOR PORE SOLUTION + 3.5 wt.% NaCl + 0.300 M $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$

Disk #	Thickness (mm)	Time (hr)	Surface	C	O	Si	Ca	Cl	B	Na
9	6.2	0.17	Top	38.4	41.3	4.8	5.7	0.3	3.0	6.5
			Bottom	44.7	39.1	7.6	6.5	0.2	0.66	1.2
10	5.6	0.5	Top	46.7	36.1	7.5	5.4	0.2	1.7	2.4
			Bottom	45.9	39.0	8.4	4.7	0.2	0.2	1.5
11	5.5	1.5	Top	36.5	43.1	6.4	6.1	0.2	3.6	4.2
			Bottom	35.6	43.9	9.9	5.8	0.2	0.8	3.7
12	6.3	25	Top	37.2	42.2	8.3	6.1	0.4	1.5	4.5
			Bottom	33.4	39.4	2.9	2.0	3.1	6.1	13.2
4	4.9	48	Top	32.7	45.0	6.3	5.3	0.4	5.4	5.0
			Bottom	39.9	37.1	1.0	0.9	3.6	5.8	11.7

TABLE 4 XPS ANALYSIS FOR SODIUM MFP MIGRATION THROUGH CONCRETE: SOLUTION PENETRATION RESULTS FOR PORE SOLUTION + 3.5 wt.% NaCl + 0.300 M MFP

Disk #	Thickness (mm)	Time (hr)	Surface	C	O	Si	Ca	Na	Cl	F	P
17	7.2	8	Top	45.7	29.5	1.6	0.8	13.9	2.1	3.1	3.3
			Bottom	39.0	41.5	10.8	6.4	1.2	0.3	0.5	0.3
20	5.8	24	Top	34.4	43.4	6.2	5.4	5.2	0.2	0.9	4.3
			Bottom	34.5	45.6	10.4	5.8	2.9	0.2	0.2	0.4
21	7.7	48	Top	31.4	44.6	4.3	7.1	5.0	0.3	0.8	6.5
			Bottom	32.8	46.5	10.1	7.2	2.4	0.3	0.3	0.4
22	6.2	72	Top	27.5	46.9	4.0	7.9	5.0	<0.2	1.3	7.4
			Bottom	41.6	42.7	8.0	5.7	1.1	0.4	0.3	0.2
23	7.2	114	Top	41.6	35.8	5.1	4.9	6.0	0.3	1.1	5.2
			Bottom	45.2	39.2	7.4	6.1	1.2	0.4	0.2	0.3

it is informative to recall the elements that exist in the inhibitor solution. The solution is composed of NaOH, NaCl, and $\text{Na}_2\text{PO}_3\text{F}$, [plus KOH and $\text{Ca}(\text{OH})_2$], yielding respective total concentrations of Na, 1.500 M; Cl, 0.600 M; P, 0.300 M; and F, 0.300 M—or the atomic ratios of Na:Cl:F:P of 5:2:1:1. Analysis of the top surface of the disk having been in contact with MFP-pore solution for 8 hr exhibits ratios of Na:Cl:F:P of 5:0.75:1.1:1.2. This result is in good agreement with the expected ratios except that the chlorine content is significantly lower than expected. Examination of the results for longer exposure times revealed that the chlorine percent remained at a level of about 0.3 ± 0.1 at.%, a level near the detection limit for chlorine. It is reasonable to suggest that chlorine as chloride is adsorbed below the surface of the disk. Following 24 hr of exposure and up to 114 hr, the concentration of sodium at the upper disk surface remains at a concentration of 5 to 6 at.%, whereas the concentration on the lower surface is in the range 1 to 3 at.%. If it is recognized that sodium from NaCl and NaOH represents 60 at.% of the total sodium present, and if the initial sodium content for the 8-hr top surface is taken as representative of the concentration for sodium for solute that has not migrated sufficiently into the disk, then the expected concentration for sodium if it had penetrated the disk at longer times would exceed 8 at.%. That the sodium content on the lower disk surface is 1 to 3 at.%, indicates that sodium has not penetrated the disks in these experiments.

The concentration of phosphorus on the top disk surface remains at about 6 ± 1 at.%, whereas that on the lower portion of the disk is 0.3 ± 0.1 at.%. The concentration of fluorine does not change significantly on the lower disk surfaces. These data indicate that phosphorus and fluorine and thus MFP do not appear to migrate through the disks under the present experimental conditions.

The surface analytical results for MFP elements indicate that at early times the P:F ratio was the expected 1:1, within experimental error. On the other hand, the P:F ratio on the upper surface does not remain at 1:1 for exposure times beyond 8 hr. The P:F ratio is in the range of 4.7 to 8.1, with an

average value of 5.8 for four samples. This result indicates that chemical alterations must be occurring for MFP as a result of its interaction in pore solution with concrete.

The photoelectron spectra in the F 1s region for frozen (-150°C) MFP (0.300 M) pore solution and for the top portions of disks following migration experiments are shown in Figure 3. For MFP dissolved in pore solution, only one fluorine photopeak is detected; with F 1s BE of 687.5 eV. This BE value is consistent with values measured for fluorine bonded to phosphorus in other fluorophosphates (9). The F 1s photoelectron spectrum for the top portion of a disk following MFP interaction with concrete for 8 hr reveals two fluorine peaks with BE values at 688.0 and 685.3 eV. The photopeak of higher BE value is associated with fluorine attached to phosphorus in MFP. The fluorine photopeak at the lower binding energy is assigned to fluoride as in metal fluorides such as sodium or as adsorbed fluoride (9). In the fluorine spectra for concrete specimens that had been exposed to MFP for longer time periods (24 and 72 hr, see Figure 3), only one F 1s peak is noted, with $\text{BE} \approx 685$ eV. These findings support the notion that interaction of MFP with concrete leads to chemical changes in MFP and that the changes occur within 24 hr. The surface analysis results are consistent with the formation of fluoride and phosphate species in the hydrolysis reaction. Assuming that fluoride is produced, the surface analytical results, which show a greater concentration of phosphorus on the disk surface, indicate that fluoride is not as strongly adsorbed on the concrete surface as is phosphate or

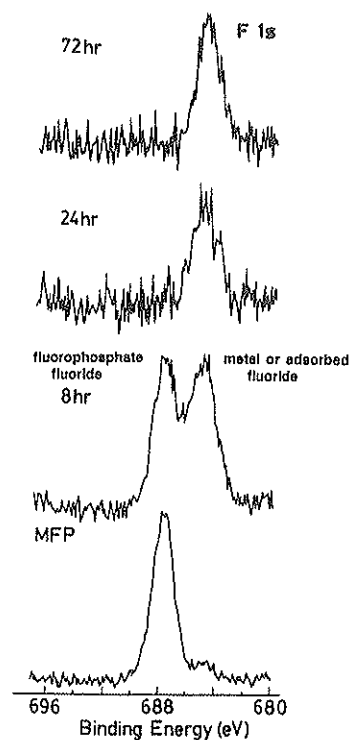


FIGURE 3 F 1s photoelectron spectra, illustrating the change in fluorine chemistry that takes place as a result of interaction with the concrete disk.

that fluoride diffuses into the concrete disk at a faster rate than phosphate. These findings demonstrate that MFP itself may not be an effective inhibitor, but that its hydrolysis products may be the active species delivered at the rebar-concrete interface.

CONCLUSIONS

The migration of inhibitor constituents through concrete disks has been studied, and the migration varied with the square root of time. The migration time was determined by measuring the change in volume for solutions in contact with concrete disks while one side of the disk was maintained under vacuum. Analysis of the disk surfaces was used also to measure the time required to penetrate the disks. The chemical nature of the emerging solute was evaluated with respect to elemental concentrations and chemical form from BE measurements using photoelectron spectroscopy. The transport of sodium tetraborate through the disk resulted in no chemical change in the inhibitor. Migration of sodium MFP, however, led to hydrolysis of the salt. It is suggested that the hydrolysis of MFP produces fluoride ion and phosphate species in solution. The effect of these ions on the integrity and strength of concrete was not investigated.

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DISCUSSION

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The authors present the results of a study of the penetration of various ionic species in a simulated pore solution, through 7-day cured mortar. Sodium monofluorophosphate (MFP) was included in this study. The results failed to indicate any penetration of MFP through the mortar disks. It is known that MFP will hydrolyze in alkaline solution that contains an excess of calcium hydroxide. The experimental conditions used in this study were precisely alkaline solutions, saturated with calcium hydroxide, forced through freshly made mortar. These are the best conditions that cause hydrolysis of MFP.

The results presented in the paper clearly demonstrate that MFP did not penetrate the mortar under these conditions. Furthermore, the mortar prevented the penetration of sodium and chloride ions because of the blocking effect of the hydrolysis products of MFP. This property should have a positive effect on corrosion prevention of reinforcing steel in concrete. Unfortunately, the volume of solution that passed through the mortar disks for the MFP experiment is not reported.

However, the conditions of this study do not reflect the conditions encountered in aged concrete. In this case, many published studies have shown that the pore solution contains a limited amount of calcium hydroxide; the concrete might also be carbonated, reducing its alkalinity. Hydrolysis of MFP is therefore reduced or eliminated in old concrete. Partial drying of the concrete (naturally or artificially) also greatly enhances the penetration of an ionic species in solution. In field treatment of a structure with a corrosion inhibitor, neutral solutions should also be used, again diminishing the possibility of hydrolysis for MFP.

In many laboratory experiments, it has been demonstrated that MFP can easily penetrate through concrete at a depth of 3 cm or more. Multiple treatment with partial drying of concrete also enhances the penetration.

AUTHORS' CLOSURE

We appreciate the comments of Mr. Beaudoin. In this investigation, it was our objective to study the interaction of solute species under chemical conditions similar to those in concrete—thus we used Ca^{2+} saturated pore solution. The results demonstrate that monofluorophosphate (MFP) hydrolyzes under these experimental conditions. We have not studied nor have we determined or demonstrated specifically whether or not the hydrolysis product(s) or MFP itself produces a blocking effect. We have only demonstrated that under the experimental conditions, MFP hydrolyzes, producing at the concrete surface fluoride species attributed to fluoride associated with metal ions or adsorbed fluoride. The time variation of the XPS spectral features also demonstrates that, under our experimental conditions, the hydrolysis reaction is not instantaneous.

In addition, it is known that pore solution of aged concrete is a saturated calcium hydroxide solution along with potassium and sodium hydroxides. Also, as cement continues to hydrate,

calcium hydroxide is produced along with calcium silicate hydrates. Under normal field conditions, concrete in a temperate environment, such as the northeast portion of the United States, does not dry out to any significant degree below a depth of 10 mm. Concrete in the United States does not carbonate over a depth of 10 mm in 50 years because of the relatively low water-cement ratios that are used. Thus, hy-

drolysis of MFP in concrete under the field conditions described would occur.

The opinions, findings, and conclusions reported herein are those of the authors and not necessarily those of the sponsoring agency.

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