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**PHOSPHATE IONS – DOES EXPOSURE LEAD TO  
DEGRADATION OF CEMENTITIOUS MATERIALS?**

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**ABSTRACT**

An assessment of the potential effects of phosphate ions on cementitious materials was made through a review of the literature, contacts with concrete research personnel, and conduct of a “bench-scale” laboratory investigation [1]. The objectives of this limited study were to: (1) review the potential for degradation of cementitious materials due to exposure to high concentrations of phosphate ions; (2) provide an improved understanding of any significant factors that may lead to a requirement to establish exposure limits for concrete structures exposed to soils or ground waters containing high levels of phosphate ions; (3) recommend, as appropriate, whether a limitation on phosphate ion concentration in soils or ground water is required to avoid degradation of concrete structures; and (4) provide a “primer” on factors that can affect the durability of concrete materials and structures in nuclear power plants. Results of a literature review, contacts with industry personnel, and a laboratory investigation indicate that no harmful interactions occur between phosphate ions and cementitious materials unless phosphates are present in form of phosphoric acid. Relative to the “primer,” NUREG/CR-6927, published in February 2007, provides a review of pertinent factors that can affect the durability of nuclear power plant reinforced concrete structures.

**1. INTRODUCTION**

The Generic Aging Lessons Learned Report (GALL), NUREG-1801, specifies limits for sulfate and chloride ion concentrations in ground water and soil environments to avoid degradation of concrete structures. During presentation of the results of a review of a nuclear power plant license renewal application to the Advisory Committee on Reactor Safeguards,

the U.S. Nuclear Regulatory Commission Staff was asked why a quantitative allowable phosphate limit is not included as part of the GALL acceptance criteria for defining aggressive ground water/soil environments in license renewal application reviews. The Staff's response was that the current criteria for defining aggressive ground water/soil environments are primarily based on American Concrete Institute (ACI) standards and industry expert's opinion and the current criteria are considered satisfactory; however, the Staff committed to take an in-depth review of the issue.

**2. THERMODYNAMIC AND KINETIC RESULTS**

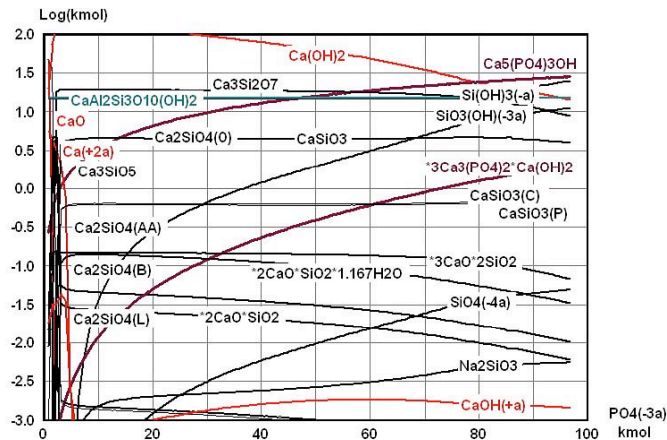
Thermodynamic calculations were performed at ORNL using the thermodynamic database in Outokumpu's HSC Chemistry V5.11 code [2]. These calculations indicate that phosphate could replace calcium hydroxide  $[\text{Ca}(\text{OH})_2]$  with calcium hydroxylapatite  $[\text{Ca}_5(\text{PO}_4)_3\text{OH}]$ . In Table 1 these data show a very strong tendency for the formation of calcium hydroxylapatite. The negative  $\Delta G$ 's ( $< -42$  kcal/mole) resulting in very large equilibrium coefficients ( $K > 10^{33}$ ) show that this reaction is spontaneous and that the reaction equilibrium goes from left to right as written in Table 1 (calcium hydroxylapatite density of  $\sim 3.08$  gm/ml). Compared to the conversion of calcium hydroxide to calcite ( $\text{CaCO}_3$ ), which naturally occurs in ordinary Portland cement's (OPC's) exposed to air and ground waters and results in a  $\sim 4\%$  volume increase, the formation of phosphates results in a net volume decrease of 3.3% compared to the calcium hydroxide conversion (i.e., 3.87% increase vs 4% increase).

Since the reaction in Table 1 only describes the reactions of calcium hydroxide and phosphate ions in an isolated system,

**Table 1.** Phosphate Replacement of Ca(OH)<sub>2</sub> in Ordinary Portland cements (OPC).

$5\text{Ca}(\text{OH})_2 + 3\text{PO}_4(-3a) = \text{Ca}_5(\text{PO}_4)_3\text{OH} + 9\text{OH}(-a)$					
T (°C)	ΔH (kcal)	ΔS (cal/K)	ΔG (kcal)	K	Log(K)
0	-7.725	127.84	-42.64	1.33E+34	34.122
10	-7.647	128.11	-43.92	8.03E+33	33.904
20	-7.391	129.00	-45.21	5.08E+33	33.706
30	-7.016	130.26	-46.50	3.38E+33	33.529
40	-6.563	131.73	-47.81	2.36E+33	33.372
50	-6.054	133.33	-49.14	1.72E+33	33.236
60	-5.497	135.02	-50.48	1.31E+33	33.118
70	-4.901	136.79	-51.84	1.05E+33	33.019
80	-4.271	138.60	-53.22	8.63E+32	32.936
90	-3.612	140.44	-54.61	7.39E+32	32.869
Formula	FM (g/mole)	Conc. (wt-%)	Amt. (mol)	Amt. (g)	Vol. (ml)
Ca(OH) <sub>2</sub>	74.095	56.527	5	370.473	165.39
PO <sub>4</sub> (-3a)	94.971	43.473	3	284.914	0
Ca <sub>5</sub> (PO <sub>4</sub> ) <sub>3</sub> OH	502.32	76.645	1	502.321	159.98
OH(-a)	17.007	23.355	9	153.066	0
				<b>Volume Change =</b>	<b>-3.3%</b>

we looked to see if this strong reaction could compete with the calcium aluminosilicates that are formed in OPC matrices. Figure 1 shows the equilibrium phases for an OPC system that is inundated with phosphate ions. These data show that in an OPC system the formation of calcium hydroxylapatite is still capable of replacing the free calcium hydroxide (Portlandite) and competes successfully for calcium in aluminosilicate matrices.



**Figure 1** Phases in OPC that form under increasing exposure to phosphate.

Kinetic results presented in the literature [3-5] indicate that in high or medium supersaturated solutions formation of hydroxylapatite always seems to be preceded by the precipitation of one or more precursor phases. Homogeneous formation of hydroxylapatite even at low supersaturation was never seen.

### 3. LITERATURE REVIEW/INDUSTRY CONTACTS

A review of the literature did not identify any pertinent information relative to harmful interactions of phosphate ions and cementitious materials. Where phosphate was present and degradation of concrete occurred, this was generally due to phosphate being in the form of phosphoric acid which is known to cause degradation of Portland cement-based materials [6,7]. Since natural water systems are buffered by carbonates, aluminates, and silicates, phosphate ions resulting from sources such as fertilizer will not lower the pH sufficiently to produce H<sub>3</sub>PO<sub>4</sub> as a predominant species. Also, the presence of Ca and Mg will precipitate the PO<sub>4</sub> to further reduce the possibility that an acid environment can persist. Phosphate compounds have been included in concrete mixes as set retarders. A number of cement-based binders or phosphate cements have been produced for civil-engineering repair applications to take advantage of their quick setting capability. Several of the articles addressed apatite and dental applications. Finally, phosphogypsum, a main by-product of the phosphate industry, has been evaluated for application as a road base material and set retarder for Portland cement concretes [8].

Numerous contacts with cognizant research personnel and related industry organizations did not identify any apparent concerns or incidences of deterioration of reinforced concrete structures located in phosphate-rich locations. No special design requirements (e.g., increased cover, concrete mix proportions, or materials selection) were identified for concrete structures located in high-phosphate environments similar to that which exists for reinforced concrete structures in high sulfate or chloride environments. Finally, contacts with

organizations involved with reinforced concrete structures located in high phosphate regions of Florida has not revealed any concerns or evidence of degradation of these structures due to exposure to phosphates contained in soils or ground water.

#### 4. LABORATORY INVESTIGATION

Thermodynamic studies and kinetic results presented earlier were utilized to design and implement a “small-scale” laboratory study. The laboratory study was designed to investigate the effects of two aqueous phosphate systems representing different phosphate solubilities (i.e.,  $\text{Na}_2\text{HPO}_4$  and  $\text{MgHPO}_4$ ) on a Portland cement paste. The effect of the phosphate systems was assessed through comparisons of weight change, length change, and compressive strength results to those obtained from specimens exposed to a control environment [i.e., saturated  $\text{Ca}(\text{OH})_2$  solution]. X-ray diffraction and scanning-electron microscope examinations were performed on materials obtained from representative specimens after being subjected for defined periods to each of the conditioning environments. The purpose of these supplemental examinations was to identify formation of any significant new phases or to determine if the specimens show signs of degradation or secondary mineralization, phosphate minerals in particular.

#### 4.1 Specimen Preparation and Conditioning

Type I-II Portland cement paste having a ratio of water to cement of  $\sim 0.30$  was used to fabricate the test articles. Prismatic length-change ( $25.4 \times 25.4 \times 254.0\text{-mm}^3$ ) and cube specimens ( $50.8\text{-mm}^3$ ) were fabricated from the mix. One-day after casting, the test articles were removed from their molds and cured under saturated conditions (100% relative humidity) until exposure conditioning initiated.

Conditioning of the test articles involved three exposure solutions: two concentrations of phosphate ions and a control solution. The phosphate solutions were selected to provide concentrations of phosphate ions much higher than normally experienced in service and included: (1) a saturated low-soluble phosphate salt, magnesium phosphate ( $\text{MgHPO}_4$ ) and (2) a saturated high-solubility phosphate salt, sodium hydrogen phosphate dodecahydrate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ). A saturated calcium hydroxide solution was utilized as the control for comparison of the phosphate results.

Excess solids of these salts were poured onto the bottom of plastic trays containing sufficient water that the cubes and prisms were completely submerged. The aqueous brine solutions became saturated in equilibrium with the excess solids. As a result, if the cement extracted phosphate from the brines, constant concentrations in the exposure brines were maintained during the concrete (cement paste) interaction with the brine. The test specimens were suspended above the excess solids on the bottom of the conditioning trays by using polyvinylchloride (PVC) corner braces.

#### 4.2 Specimen Testing

After predefined conditioning periods of 30-days, 3-months, 6-months, 1-year, and 18-months, specimens were removed from each of the conditioning solutions and tested. Tests performed after each of the conditioning periods included length change, weight change, and compressive strength. In addition, aliquots of representative materials were examined by x-ray diffraction (XRD) and scanning-electron microscope (SEM) techniques.

The results obtained for length change are summarized in Figure 2. After 18 months exposure, the length increase for the  $\text{Na}_2\text{HPO}_4$  was measured to be around 11 percent while the increase for the  $\text{Ca}(\text{OH})_2$  and  $\text{MgHPO}_4$  was found to be around 15 percent. The increase in length observed for all the specimens was probably caused by an expansion created by carbonates developing inside the cement paste matrix.

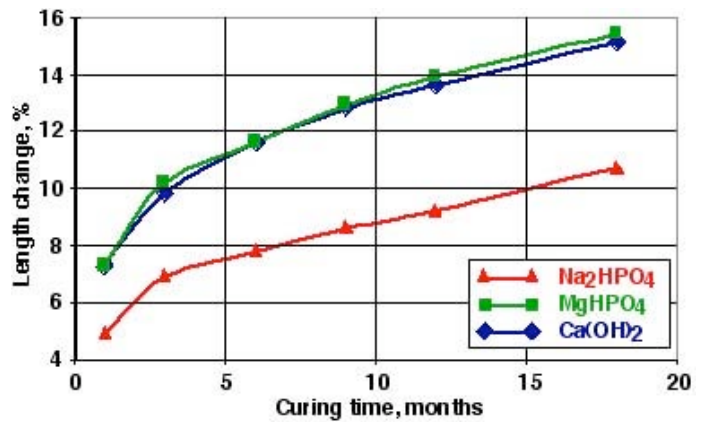


Figure 2 Length change test results.

The results obtained for weight change are summarized in Figure 3. The weight change for the specimens conditioned in the phosphate solutions is less than that obtained for specimens conditioned in the reference  $\text{Ca}(\text{OH})_2$  solution. The results obtained for the specimens conditioned in the  $\text{MgHPO}_4$  solution tended to also form carbonate crystals. The larger gain of

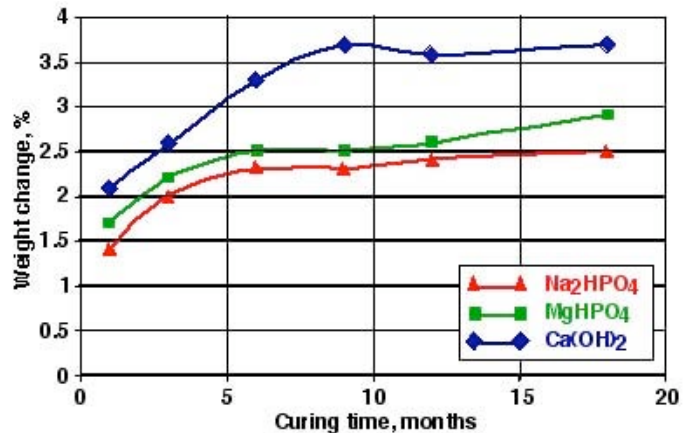


Figure 3 Weight change test results.

weight for the samples kept in the  $\text{Ca}(\text{OH})_2$  solution is probably due to the large amount of calcium carbonate deposits present on the surfaces of the specimens. The variation observed on the chart is confirmed by the visual observations of carbonate growth on the specimen's surfaces.

The effect of curing conditions on compressive strength of 50.8-mm<sup>3</sup> test specimens is presented in Figure 4. Three specimens were tested for each solution at each curing time presented in the figure. For some results, primarily early in the test program, the error bar is wide; this is usually caused when a defect is present in one specimen (e.g., air bubble). Due to the limited number of tests for each curing solution at each of the curing times, a statistical evaluation of the measurements did not allow removing any measured value as an outlier. As noted in the figure, the compressive strength values obtained after 18 months cure were high, between 16,000 and 20,000 psi (110 to 138 MPa) for specimens cured in each of the three solutions (MPa = psi x .006894757). The figure shows that the compressive strengths have generally increased since the beginning of the test, 18 months ago. If the phosphate solutions were having a deleterious affect on the cement paste specimens, it would be expected that the compressive strength would decrease with increasing exposure period, which is opposite to what has been observed.

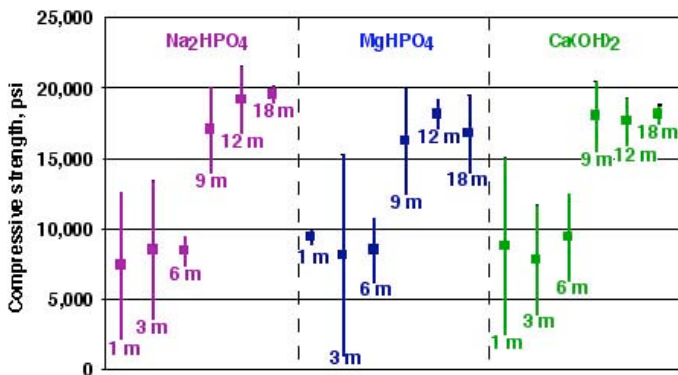


Figure 4 Compressive strength test results.

Two x-ray diffraction (XRD) samples per specimen were prepared for each curing condition. The first sample was collected from the outer surface of the specimen by scraping the surface with a scalpel. The thickness of this layer is estimated to be approximately two hundred microns. The second sample was also obtained near the outside layer of the prism, but had a thickness of about 5-8 mm. A comparison of the spectra obtained at the two depths is used to determine if penetration of the phosphate salts occurred to create new mineral phases. The spectra obtained from the four samples cured in the phosphate solutions are shown in Figure 5. As one can see, the spectra contain many peaks with most of them overlapping thus making identification of the mineral phases present difficult. The main mineral phases identified in all four spectra are: Ettringite ( $\text{Ca}_6(\text{Al}(\text{OH})_6)_2(\text{SO}_4)_3(\text{H}_2\text{O})_{25.7}$ ),

Portlandite ( $\text{Ca}(\text{OH})_2$ ), Calcite ( $\text{CaCO}_3$ ), Brownmillerite [ $\text{Ca}_2(\text{Al}, \text{Fe}^{3+})_2\text{O}_5$ ], and Larnite ( $\text{Ca}_2\text{SiO}_4$ ). The peaks for hydroxylapatite [ $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ ] and carbonatehydroxylapatite [ $\text{Ca}_{10}(\text{PO}_4)_3(\text{CO}_3)(\text{OH})_2$ ] cannot be identified with certainty. The spectra obtained for the first 200 microns (external zone) show that calcite is very abundant, as would be expected in this layer, while Portlandite is less abundant than identified in the outside layers of the samples (external layer).

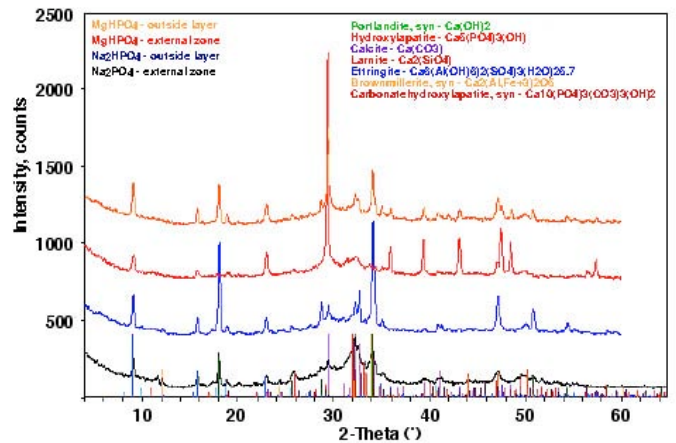


Figure 5 XRD spectrum from samples cured in phosphate solutions.

Scanning-electron microscope (SEM) examinations performed on specimens after 18 months of conditioning in each of the solutions confirmed the results found by x-ray diffraction. As noted in Figure 6, the cement paste is very dense for the specimen cured in  $\text{Na}_2\text{HPO}_4$ . The outside layer does not

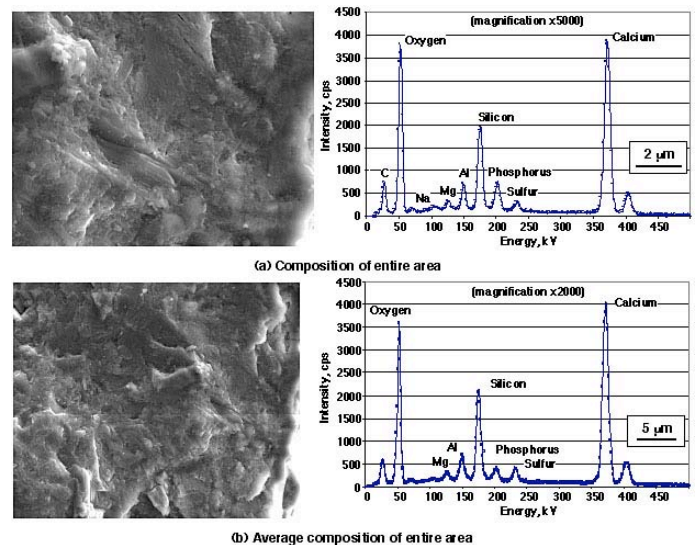


Figure 6 SEM of outside layer of sample cured in  $\text{Na}_2\text{HPO}_4$  solution.

exhibit much porosity. Crystals of Portlandite and sulfo-aluminates are visible in the figure. Phosphorus is present as seen on the energy-dispersive x-ray spectroscopy (EDX) spectrum.

Crystal forms of hydroxylapatite are difficult to identify in the dense paste and are probably finely mixed with the calcium-silicate-hydrate (C-S-H); however, locally, as shown in Figure 7, one can find small areas that possibly contain minerals belonging to the hydroxylapatite family.

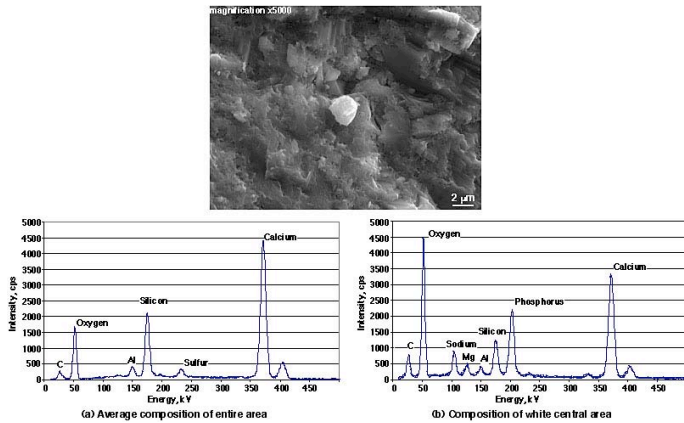


Figure 7 Crystal containing calcium and phosphorus.

The border of the sample was examined in detail, but large formations of calcium phosphate were not found. It appears that the phosphorus present is mixed with other crystalline phases, probably the C-S-H as indicated elsewhere [9]. Although rare, a well-crystallized calcium phosphate phase is seen inside a cement paste pore at the outside surface of the sample as shown in Figure 8.

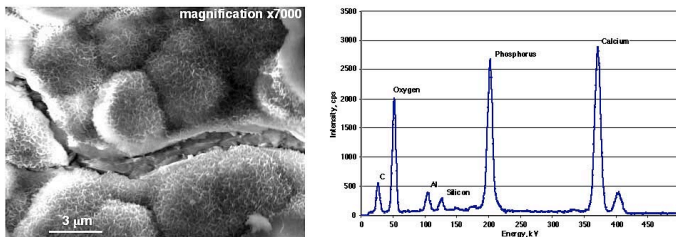


Figure 8 Crystals of calcium phosphate (possibly hydroxylapatite) found in cement pore near outside surface.

### 4.3 Conclusions After Eighteen-Months Exposure

After eighteen months of curing in saturated solutions of  $\text{Na}_2\text{HPO}_4$ ,  $\text{MgHPO}_4$  or  $\text{Ca}(\text{OH})_2$  (baseline), the cement paste specimens do not show any sign of deleterious reactions such as cracking and spalling. The specimens cured in the  $\text{Ca}(\text{OH})_2$  solution are covered with carbonate crystals, but the specimens cured in  $\text{Na}_2\text{HPO}_4$  are free of any deposits. The physical testing showed a decrease in length expansion for the specimens cured in the  $\text{Na}_2\text{HPO}_4$  solution when compared to the specimens cured in  $\text{Ca}(\text{OH})_2$  while the specimens cured in the  $\text{MgHPO}_4$  solution exhibited the same expansion as the reference  $\text{Ca}(\text{OH})_2$  solution. Weight changes exhibited the same pattern. Compressive strength results obtained were

uniformly high [16,000 to 20,000 psi (110 to 138 MPa)], independent of the particular curing solution. Results indicate no significant deleterious reactions resulting from up to 18-months cure in concentrations of phosphate ions much higher than would be normally experienced in service.

Analyses by XRD and SEM showed the presence of calcium phosphate (probably hydroxylapatite and carbonate-hydroxylapatite) in the samples cured in phosphate solutions. The results from the thermodynamic calculations were predicting that the final hydration product would be hydroxylapatite and the tests performed showed the presence of this phase. Hydroxylapatite is very stable and highly insoluble; therefore, the durability of cement or concrete where insoluble hydroxylapatite has replaced the slightly soluble calcium hydroxide should equal or increase the concrete durability.

## 5. PRIMER ON DURABILITY

As a part of the overall program, a primer on the environmental effects that can affect the durability of nuclear power plant concrete structures was prepared [10]. Provided below is an abstract of the report.

As concrete ages, changes in its properties will occur as a result of continuing microstructural changes (i.e., slow hydration, crystallization of amorphous constituents, and reactions between cement paste and aggregates), as well as environmental influences. These changes do not have to be detrimental to the point that concrete will not be able to meet its performance requirements. Concrete, however, can suffer undesirable changes with time because of improper specifications, a violation of specifications, or adverse performance of its cement paste matrix or aggregate constituents under either physical or chemical attack. Contained in this report is a discussion on concrete durability and the relationship between durability and performance, a review of the historical perspective related to concrete and longevity, a description of the basic materials that comprise reinforced concrete, and information on the environmental factors that can affect the performance of nuclear power plant concrete structures. Commentary is provided on the importance of an aging management program. Also included in the report are descriptions of safety-related nuclear power plant structures, a review of operating experience for nuclear power plant concrete structures, and a discussion on the relationship between concrete cracking and the corrosion of embedded steel reinforcement.

## 6. SUMMARY AND CONCLUSIONS

The objectives of this limited study were to: (1) review the potential for degradation of cementitious materials due to exposure to high concentrations of phosphate ions; (2) provide an improved understanding of any significant factors that may lead to a requirement to establish exposure limits for concrete structures exposed to soils or ground waters containing high levels of phosphate ions; (3) recommend, as appropriate,

whether a limitation on phosphate ion concentration in soils or ground water is required to avoid degradation of concrete structures; and (4) provide a “primer” on factors that can affect the durability of concrete materials and structures in nuclear power plants. An assessment of the potential affects of phosphate ions on cementitious materials was made through conduct of thermodynamic calculations and a review of available kinetic results, a review of the literature, contacts with concrete research personnel, and conduct of a “bench-scale” laboratory investigation.

Thermodynamic calculations performed using Outokumpu’s HSC Chemistry V5.11 code indicate that phosphate could replace calcium hydroxide  $[\text{Ca}(\text{OH})_2]$  with calcium hydroxylapatite  $[\text{Ca}_5(\text{PO}_4)_3\text{OH}]$ . Kinetic results presented in the literature indicate that in high or medium supersaturated solutions, formation of hydroxylapatite always seems to be preceded by the precipitation of one or more precursor phases. Homogeneous formation of hydroxylapatite even at low supersaturation was never seen.

A review of the literature did not identify any pertinent information relative to interaction of phosphate ions and cementitious materials. Where phosphate was present and degradation of concrete occurred, this was generally due to phosphate being in the form of phosphoric acid which is known to cause degradation of Portland cement-based materials. Phosphate compounds have been included in concrete mixes as set retarders. A number of cement-based binders or phosphate cements have been produced for civil-engineering repair applications to take advantage of their quick setting capability. Several of the articles addressed apatite and dental applications. Finally, phosphogypsum, a main by-product of the phosphate industry, has been evaluated for application as a road base material and set retarder for Portland cement concretes.

Numerous contacts with cognizant research personnel and related industry organizations did not identify any apparent concerns or incidences of deterioration of reinforced concrete structures located in phosphate-rich locations. No special design requirements (e.g., increased cover, concrete mix proportions, or materials selection) were identified for concrete structures located in high-phosphate environments similar to that which exist for reinforced concrete structures in high sulfate or chloride environments. Finally, contacts with organizations involved with reinforced concrete structures located in high phosphate regions of Florida have not revealed any concerns or evidence of degradation of these structures due to exposure to phosphates contained in soils or ground water.

After eighteen months of curing in saturated solutions of  $\text{Na}_2\text{HPO}_4$ ,  $\text{MgHPO}_4$  or  $\text{Ca}(\text{OH})_2$  (baseline), none of the cement paste specimens showed any sign of deleterious reactions such as cracking and spalling. Length and weight change data for the specimens cured in the phosphate solutions did not exhibit any significant deviation from data obtained from baseline

specimens cured in the  $\text{Ca}(\text{OH})_2$  solution (i.e., after 18-months curing the length and weight change exhibited by specimens cured in the phosphate solutions was comparable to or less than that obtained from the control solution). Results obtained for compressive strengths were uniformly high [16,000 to 20,000 psi (110 to 138 MPa)], independent of the particular curing solution. No significant difference was observed between results obtained from specimens cured in the phosphate solutions and the specimens cured in the  $\text{Ca}(\text{OH})_2$  solution (baseline).

Analyses by XRD and SEM showed the presence of calcium phosphate (probably hydroxylapatite and carbonate-hydroxylapatite) in the samples cured in the phosphate solutions. The results from the thermodynamic calculations were predicting that the final hydration product would be hydroxylapatite and the tests performed showed the presence of this phase. Hydroxylapatite is very stable and highly insoluble; therefore, the durability of cement or concrete where insoluble hydroxylapatite has replaced the slightly soluble calcium hydroxide should equal or increase the concrete durability.

In conclusion, results of this investigation indicate that: no harmful interactions occur between phosphates and cementitious materials unless phosphates are present in the form of phosphoric acid; phosphates have been incorporated into concrete as set retarders, and phosphate cements have been used for infrastructure repair; no standards or guidelines exist pertaining to applications of reinforced concrete structures in high-phosphate environments; interactions of phosphate ions and cementitious materials has not been a concern of the research community; and laboratory results indicate similar performance of specimens cured in phosphate solutions and those cured in a calcium hydroxide solution after exposure periods of up to eighteen months.

## 7. DISCLAIMER

This work was performed under the auspices of the United States Nuclear Regulatory Commission (USNRC) and was prepared in part by an employee of the USNRC. The USNRC has neither approved nor disapproved its technical content. The paper presents information that does not currently represent an agreed-upon staff position. Neither the United States Government nor any agency thereof, nor any employee, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for any third party’s use, or the results of such use, of any information, apparatus, product, or process disclosed in this publication, or represents that its use by such third party would not infringe privately owned rights.

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