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Un-reacted Metasilicate Contamination in Hardened Concrete

The application of topical curing compounds and surface hardeners to hardened concrete floor slabs represents a common construction practice employed throughout much of North America. The majority of these products are comprised of sodium and/or potassium-rich metasilicates (comparable to water glass). The application of these materials to un-cured concrete serves to stimulate a chemical reaction whereby crystals of portlandite [$\text{Ca}(\text{OH})_2$] contained in the cement paste combine with the Na or K-rich metasilicate to form calcium silicate hydrate. This 'chemical-flood' of metasilicate contributes to an increased intergrowth of the cement paste constituents within the uppermost 5–20 mm of the concrete surface, thereby reducing the interconnectivity or permeability of the micropores of the cement matrix, and trapping minute particles and films of un-reacted metasilicate residue. Factors that influence the concentration & penetration depth of the un-reacted metasilicate residues include: 1) the relative abundance of portlandite, 2) the porosity & permeability of the concrete, and 3) the relative concentration of metasilicate applied. The residual metasilicates are hygroscopic & likely include loosely attached hydroxyl radicals & water molecules. In the presence of adequate concentrations of moisture vapor, the superposition of an impermeable floor coating system or membrane can result in the gradual accumulation of a condensate layer immediately beneath the membrane. For concrete slabs treated with these metasilicate products, the localized dissolution of the metasilicate residues and reaction byproducts results in the rapid development of an ionic concentration gradient within the condensate layer, thus providing a significant driving force for osmosis. The process of osmosis can be characterized as nature's effort to balance the concentration disparity between the condensate layer and the moisture vapor occupying the pore system throughout the underlying concrete slab. Within the context of an osmotic cell, the transmission of moisture vapor occurs in the direction of the fluid layer characterized by relatively elevated ionic concentrations, thus serving to dilute the more concentrated (condensate) solution.

Given the deleterious effects of the metasilicate residues & osmosis on the adhesion of impermeable floor coating systems, the problem becomes one of developing an analytical strategy capable of identifying the presence of the un-reacted, Na &/or K-rich metasilicate residues & byproducts. This analytical challenge is magnified by the nature of the solid chemistry of the concrete that is characterized by significant (and somewhat variable) proportions of silicon, sodium and potassium. Research conducted at Mineralogy, Inc. on hardened concrete test specimens treated with Na-rich metasilicate curing compounds indicates that these samples exhibit consistently elevated levels of water-soluble Na and K ions within the uppermost surface layers of the test samples, relative to control (un-treated) concrete specimens. This research has contributed to the development of a testing protocol specifically designed to evaluate the concentration of water-soluble Na & K ions (within the uppermost surface horizons of concrete core samples) via ion chromatography. All concrete (treated & un-treated) contains some degree of water-soluble Na and K ions. By applying the ion chromatography testing methodology to many hundreds of concrete core samples (at depths well below the potential influence of topically applied metasilicate products), we have acquired a database of analytical values that establish 'normal, water-soluble Na & K ion concentration windows' for untreated concrete, permitting us the ability to recognize 'water-soluble ion anomalies' for concretes with indeterminate treatment histories.

Ion Chromatography (IC) Analysis

The purpose of the ion chromatography test method is to assess the concentration levels of water-soluble ionic phases that are associated with contaminants that are known to contribute to the delamination of vapor barriers applied to concrete. These contaminants include: Na &/or K-rich metasilicate residues & byproducts (commonly applied as curing compounds, surface densifiers, & hardeners), sulfate-rich surface precipitates, and excess chloride salts. Two discrete profile positions [0-3 mm & 3-5 mm BTC (Below the Top of the Core)] are commonly evaluated using the ion chromatography technique. The IC test assesses the proportion of selected water-soluble ions (Na, K, SO₄, and Cl) in the concrete. The ionic concentrations for the 3-5 mm BTC sample provide a sense of the concentration gradient or penetration depth for the selected water-soluble ions, relative to the uppermost profile slice of the concrete (0-3 mm BTC). At Mineralogy, Inc., we have applied this testing protocol to over 1000 core samples from across the US. The data set for the first 600 specimens tested included an evaluation of the same ion concentrations (Na, K, SO₄, and Cl) for concrete collected from the 25-30 mm BTC slice (well removed from the influence of topically applied contaminant sources). Based on this database of IC concentration data, normal concentration ranges for these water-soluble ions are noted as follows:

Sodium (Na) ~200-800 ppm
Potassium (K) ~200-800 ppm
Chloride (Cl) ~10-100 ppm
Sulfate (SO₄) ~1500-5500 ppm

When the IC data set collected for an individual core sample exhibits Na &/or K ion values that are > 1000-1200 ppm, our research indicates that the presence of metasilicate gel residues constitute a likely source for the anomaly. Thin section petrography and/or x-ray diffraction analysis are routinely recommended for core samples that exhibit elevated levels of water-soluble potassium, sulfate and/or chloride ion values, as these anomalies can signal other potential problems (aside from contamination related to the presence of un-reacted metasilicate residues and byproducts) with the concrete (e.g., ASR and sulfate degradation).

The identification of un-reacted metasilicate residues and byproducts within depth specific profile slices of concrete core material, allows for the implementation of a floor preparation protocol designed to remove the contaminated cement paste (prior to the application of the vapor membrane), thereby reducing the occurrence of osmosis (& flooring system failures). In this case, knowledge equates to power & the ability to forestall or circumvent the occurrence of an expensive flooring system failure. The science is clear that un-reacted metasilicate contaminants (attributed to the application of curing compounds and/or concrete surface hardeners) contribute to the increased occurrence of osmosis & the subsequent delamination of high density, polymer flooring systems. The implementation of the IC testing methodology as performed at Mineralogy, Inc. can facilitate the identification of un-reacted metasilicate residues and byproducts within concrete floor slabs, thus contributing to the prevention of costly flooring system failures.

Mineralogy, Inc. is a geological & geotechnical testing laboratory in business since 1979, specializing in the analysis and characterization of natural and synthetic solid materials. The laboratory has participated in the analysis of over 1000 concrete core samples from across the US and Canada, specializing in the evaluation of mineralogical and chemical factors that can contribute to accelerated rates of moisture transmission and the cohesive degradation of hardened concrete.