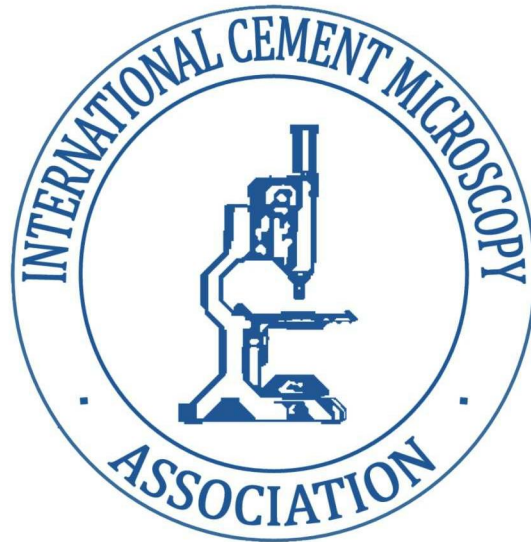


**31st INTERNATIONAL CONFERENCE
ON
CEMENT MICROSCOPY**



**April 26 - 29, 2009
St. Petersburg, FL**

NANOSCALE EXPLORATION OF PHOTOCATALYTIC CONCRETES: APPLICATION OF THE GD-OES TECHNIC

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ABSTRACT

Photocatalytic concretes are new fascinating materials, they are considered for various applications in building and environmental technology ranging from façade soiling limitation to depolluting. They are also a challenging material regarding their characterization by established analytical methods. The addition of microphases of titanium dioxide distributed randomly into heterogenous materials induces scientific problems for characterization. The main challenge is to determine the distribution of TiO₂ into a complex composite material made of natural aggregates (sizing from few cm to several μm) with micrometric hydrated cement phases (C-S-H, ettringite, monosulfate, etc.).

The photo catalyst (TiO₂), added to the cement is distributed into inter granular phases and the control of the amount and the lateral and depth distribution of the titanium oxide is one of the key factors to optimize the efficiency of this material and its applications.

INTRODUCTION

Photocatalytic concretes [1] are new fascinating materials considered for various applications in building and environmental technology ranging from façade soiling limitation to depolluting. They are also a challenging material regarding their characterization by established analytical methods. The addition of nanophases of titanium oxide distributed randomly into heterogenous materials induces scientific problems for characterisation.

The main difficulty is to determine the distribution of a small amount of a powder of TiO₂ into a complex heterogeneous composite material made of natural aggregates (sizing from few cm to several μm) and micrometric hydrated cement phases (C-S-H, ettringite, monosulfate, etc.) randomly distributed, the whole material featuring a high porosity and being of course non conductive – all aspects obviously challenging for most characterization tools.

The photo catalyst (TiO₂), added to the cement is supposed to be distributed into inter granular phases. Controlling the amount and the distribution of the titanium oxide is one of the key factors to optimize the photocatalytic efficiency of this material, its price and its applications. For example, when synthetizing depolluting cements, the producer should optimize or find a compromise between the efficiency of the depollution capability and the amount of TiO₂ added into the cement.

The photocatalytic process is a surface one and so if Titanium Oxide can be distributed mainly at the top surface, the yield of depollution and the cost of the material will be optimized.

TESTING AND EXAMINATION OF STUCCO USING TESTS DESIGNED FOR CONCRETE

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ABSTRACT

Stucco, like the mortar in concrete, is generally made with portland cement, water and sand. Stucco can be tested in the same manner as concrete utilizing the same test procedures. The compressive strength, permeability, density and absorption characteristics can provide investigators with useful information as to the quality of the stucco after it has been applied in the field. Petrographic examinations of hardened stucco can provide answers as to why particular stucco applications exhibit certain physical characteristics determined or measured by laboratory testing.

This paper describes some of the routine tests and examinations performed on stucco. It recognizes that standardized tests for stucco need to be developed and suggests standardized tests and examinations used to evaluate concrete can be applied to stucco and other cement-based materials.

INTRODUCTION

Stucco is defined as a mixture of portland cement, water and sand, applied to a full thickness (ASTM C 926) – which can be taken to mean three coats: the scratch coat, brown coat and finish coat. Collectively the coats are called “stucco” plaster. ASTM C 11 defines stucco as a portland cement-aggregate plaster mix designed for use on exterior surfaces, which is synonymous with portland cement plaster. The American Concrete Institute (ACI) Publication SP-19 defines stucco as “A plaster used for coating exterior walls and other exterior surfaces of buildings.” There is no distinction between stucco and portland cement plaster. This author suggests that stucco is cement-based plaster and can be tested the same as any cement-based material.

Determining the physical characteristics of any material is necessary before reasons for adverse or unusual behavior can be determined. Excessive cracking, delamination between coats or unusual permeability are physical manifestations that are often cited as reasons for failure of the stucco system and why the material should be repaired or replaced. An understanding of the material physical properties is important in quantifying exactly what constitutes failure of a material. Quite often what is considered excessive cracking in one person’s view is not in another’s. Low strength may be considered to be detrimental by some, yet adequate by others. Failure of stucco system, aside from the most obvious complete collapse, is sometimes a subjective argument, and very few guidelines are available for evaluating stucco material when considering if a stucco application has failed or is within the realm of “Industry Standards.”

EFFECTS OF AGEING IN PORTLAND CEMENT DOMINATED TERNARY MIXED BINDER

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ABSTRACT

This investigation was performed to study the phenomenon of hydrate-sphere formation. This form of heterogeneous setting can occasionally be observed in ternary mixed binder (TMB) which are high in Portland cement and low in calcium aluminate cement and containing hemihydrates as ternary compound. In order to enable workability the ternary mixes are retarded by means of fruit acids, e.g. tartaric acid. It was observed that the phenomenon of hydrate-sphere formation can be enhanced by aging of the dry mix. Especially quick-setting mixtures are showing changes in hydration behavior with time after storage under ambient conditions.

Heat flow measurements and in-situ XRD Rietveld analyses indicated that with increasing ageing of the TMB dry mix, the dissolution of hemihydrates and the cement phases is more retarded – sometimes up to several hours – or the system is completely inhibited from hydration. With the start of hemihydrate dissolution the system begins to hydrate. Secondary gypsum may be formed and beside ettringite monosulfate phase is generated. But the formation of hydrates does not occur in a known way. Instead, beginning of hardening starts at a few centers of hydration, which continuously grow to mm or even cm sized spheres surrounded by a still liquid matrix.

Aging of the dry mix resulted in a special form of initial reaction-kinetics. Investigations of such binder mixture systems give rise to the theory that aging of the dry mix reduces the solubility of Ca ions, as a result of which a surplus of tartaric acid causes temporary inhibition of the hydraulic system.

INFLUENCE OF TEMPERATURE ON TIME DEPENDENT PHASE CONTENT OF WHITE CEMENT DURING FIRST 20 HOURS OF HYDRATION

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ABSTRACT

Heat evolution of white cement is strongly accelerated by increasing temperature (range 16°C – 37°C). The appearance of a second or even a third maximum during the main period of hydration is strongly influenced by the temperature. Highly accurate Rietveld refinements of in-situ XRD investigations evidence that sulfate depletion is the trigger for that second heat flow event. Sulfate depletion is influenced by temperature. At higher temperatures an earlier consumption of anhydrite is observed. After dissolving of the sulfate carrier soaring decrease of C₃A becomes obvious. At the same time precipitation of ettringite is increased. Sulfate, which is needed for ettringite formation, is delivered during that time from the solution. The formation of monophases (AFm) can be observed after the final precipitation of ettringite. The monophase formation is accompanied by a further dissolution of C₃A, which leads to the third heat flow event during main period. Thus availability of sulfate carriers and reactivity of C₃A are the key parameters for hydration control of the aluminate reaction during the first 20 hours of hydration. The silicate reaction – during the first 20 hours in detail the hydration of C₃S – shows no significance to the availability of the sulfate carrier. But temperature dependence of the C₃S hydration is obvious. Investigations show also no significance to the calculated chemical composition of the potential C-S-H in the paste.

MICROSCOPIC (ESEM) VISUALIZATION OF BACTERIA AND THEIR MEDIATED MINERAL PRODUCTS REQUIRED FOR THE DEVELOPMENT OF BACTERIA-BASED SELF-HEALING CONCRETE

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ABSTRACT

Objective of this study was to develop a concrete specimen preparation protocol for ESEM analysis which allows simultaneous observation of bacteria and their mineral precipitation products on crack surfaces of a novel type of bacteria-based self-healing concrete. Although concrete hydration products and bacterially mediated mineral precipitates could be visualized without any specimen preparation, this proved to be more difficult for intact bacteria. Without specimen preparation bacterial spores appeared easily recognizable, however, the active vegetative bacteria collapsed during vacuum application. The difference is due to cell-wall characteristics, as bacterial spores have thick and dense cell walls to resist physical and chemical stresses, those of vegetative cells are much more fragile. For better visualization of structurally intact vegetative cells, specimen pre-treatment by incubation for 30 min in a 2% formaldehyde solution appeared optimal, as this caused cross-linking of proteins resulting in a strong but permeable cell wall. However, formaldehyde treatment resulted in the partial dissolution of mineral precipitation products, and we concluded therefore that specimen pre-treatment is therefore less suitable for qualitative and quantitative analysis of cement hydration and mineral deposit products on crack surfaces of concrete specimen. In practice we therefore only apply formaldehyde treatment of concrete specimens if visualization of bacteria is wanted.

INTRODUCTION

Concrete with an inbuilt active crack-healing mechanism can on the long run save considerable amounts of money as it will decrease costly manual maintenance and repair efforts. One specific type of self-healing concrete in which active bacteria produce crack-filling mineral precipitates is currently being developed in our laboratory (1). The active healing agent incorporated in the concrete matrix consists of bacterial spores plus a suitable organic compound. The latter compound can be metabolized by active bacteria and converted to mineral precipitates. During the manufacture of this type of concrete bacterial spores instead of active bacteria are added to the concrete mixture for two reasons. Bacterial spores are specialized cells which are characterized by their spherical shape and thick cell wall. They represent in fact viable but dormant cells and are produced by active vegetative bacteria when environmental conditions become unfavorable for growth. These specialized cells can survive dehydration and other chemical and mechanical stresses for long periods of up to 200 years (2). Once growth conditions become favorable, spores germinate to produce metabolically active vegetative cells again. Laboratory experiments have shown that specifically the spores of specific alkali-resistant bacteria can resist concrete incorporation for longer periods, particularly when immobilized in a protective environment such as porous aggregate material

Deterioration of Concrete Structures Due to Weather Exposure - Case Study

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ABSTRACT

Exterior surfaces of concrete buildings that are about 35 years old, located in a subtropical location of the USA about 1600 m from the ocean, exhibit unusual etching and deterioration that occurred especially along the rims of lightweight aggregate particles. Based on petrography and SEM studies, the deterioration appears to be the result of acid attack that was created by a combination salty sea air and moisture from both atmosphere and rain.

Key Words: concrete, deterioration, weather exposure, acid attack

A COMBINED APPROACH OF NEW VOLUME EXPANSION MEASUREMENTS, CHEMICAL AND MICROSTRUCTURAL CHARACTERIZATION TO UNDERSTAND BASIC ASR MECHANISMS

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ABSTRACT

Aggregate reactivity with respect to alkali silica reaction (ASR) is actually a function of many factors such as alkalinity, moisture, temperature, particle size and crystallinity of reactive constituent(s). The amount of alkali necessary to initiate ASR expansion (i.e., threshold alkali) is expected to be different for each aggregate. Therefore, ASR is a chemical reaction that integrates the combined effects of temperature, alkalinity, and time. Activation energy can represent this kinetic-type combined effect as a single parameter and be useful to evaluate ASR susceptibility of aggregate. Microstructural characterization of the reaction products, monitoring test solution chemistry, and characterization of the reaction kinetics (e.g., determination of activation energy) are considered as the most fundamental approach to understand the ASR mechanisms. The ultimate goal of this research is to expand the understanding of the fundamental ASR mechanisms while at the same time measure some aggregate ASR material properties which serve as appropriate input and facilitates developing predictive model for concrete.

Some as-received common reactive aggregates were tested at different levels of alkalinity (similar to as well as lower and higher than concrete pore solution alkalinity) and temperature. The volume change as a function of time with a particular alkalinity was measured at three different temperatures (e.g., 60, 70, 80⁰C) and activation energy was calculated. Higher is the activation energy the lower is the ASR reactivity of aggregate. An inverse relationship between activation energy and alkalinity was apparent from the measured activation energies at different levels of alkalinity. The effect of Ca (OH)₂ on ASR expansion was investigated. The Na, K, Ca, and pH of the test solution were measured both before and after the tests. The detailed microstructural characterization of the reaction products was conducted by ESEM, and SEM-EDS. An interesting discussion on the effects of alkalinity and temperature and role of calcium on ASR is presented.

AN INVESTIGATION OF THE USE OF WASTE SLATE AGGREGATE IN CONCRETE

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ABSTRACT

The principal objective of this project is to evaluate the potential use of waste slate materials as an alternative aggregate in concrete and to investigate whether it is a feasible option for application in low strength concrete such as strip foundation for low rise 'one to two story' domestic housing and concrete floorings.

At different ages, concrete compressive, flexural and indirect tensile strength results together with SEM and EDAX analysis were conducted and compared for mixes with and without slate aggregates.

The results show that the use of slate aggregates reduces to a certain percentage the compressive, flexural and indirect tensile strength.

The contents of Ca in the control mixes were found consistently higher in comparison to their values in the slate mixes, which explain the differences in compressive strength between the control mix and those mixes containing slate aggregates.

Keywords: Slate aggregates in concrete, compressive strength, concrete EDAX analysis.

INVESTIGATIONS OF HIGH REACTIVE PURE CEMENT PHASES BY LOW-TEMPERATURE-SYNTHESIS METHODS AND THEIR HYDRATION BEHAVIOUR

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ABSTRACT

Pure dicalcium silicate (C_2S) and tricalcium silicate (C_3S) were synthesized by solid state reaction, sol-gel synthesis, Pechini process and combustion synthesis. The investigation of the crystallization process of C_2S showed, that by low temperature synthesis methods α'_L-C_2S crystallizes at $650^\circ C$ and can be stabilized at room temperature. The hydraulic reactivity of these low sintered α'_L-C_2S samples is similar to a C_3S and α'_L-C_2S is completely hydrated after 200 hours. C_2S sintered at higher temperatures is, despite of low particle sizes of about 100 nm, not very reactive. The investigation of the crystallization behaviour of C_3S has shown, that by all synthesis methods, sintering temperatures of $1500^\circ C$ are necessary to get pure C_3S . But despite the same heating and cooling condition different modifications were stabilized. The hydration reaction shows, that the degree of hydration by C_3S produced with the low temperature synthesis methods is higher and that different morphologies of CSH phases occur.

MODIFYING THE BOGUE CALCULATIONS FOR CEMENTS USING EDX MICROANALYSES OF CLINKERS

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ABSTRACT

The Bogue calculations are routinely used to estimate the quantities of the four major mineral phases in portland cements and clinkers for compliance with specifications, such as ASTM C 150. However, the Bogue calculations can give significantly inaccurate values, typically underestimating the alite and overestimating the belite. The primary reason for this is that the major mineral phases in production clinkers contain appreciable amounts of substituent oxides, whereas the Bogue equations assume that pure compounds exist. EDX microanalysis of polished specimens in the SEM was used to determine the true compositions of the individual phases in a clinker, including the substituent oxides. The average compositions from EDX microanalyses were used to modify the Bogue equations, and in doing so obtaining calculated phase quantities for a production cement that were in much better agreement with quantitative X-ray diffraction analysis. The possibility of using such a method for routine analysis of cement is discussed.

INTRODUCTION

The Bogue calculations [1], as those provided in ASTM C 150, are routinely used to estimate the amounts of the four major mineral phases in portland clinkers and cements. It is widely known, however, that the Bogue calculations can disagree considerably with results obtained by optical microscopy or quantitative X-ray diffraction analysis [2, 3], notably by underestimating the alite and overestimating the belite. The primary reason the calculations give inconsistent results is that the four major mineral phases in clinker contain a significant content of substituent ions, while the derivation of the Bogue equations assumes that they are pure compounds. The non-equilibrium conditions that occur during rapid cooling of clinkers also contribute to the discrepancy between the calculated phase amounts and the actual phase amounts [4].

The Bogue equations are a solution of four simultaneous linear mass balance equations with the four unknowns being the amounts of the mineral phases, C_3S , C_2S , C_3A , and C_4AF . A modification to the Bogue calculations was first described by Yamaguchi and Takagi [5], where the mass balance equations were modified to reflect estimates of the actual compositions of the four major clinker mineral phases, rather than those of the pure compounds, using classical

THE INFLUENCE OF SUPERPLASTICIZERS ON THE HYDRATION OF C₃S

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ABSTRACT

Superplasticizers are widely used to enhance the fluidity and lower the water to cement ratio of concretes. Usually very efficient superplasticizers prolong the induction period of hydration and thus the setting of concretes. A basic understanding of the underlying mechanism is still being discussed. Results of the present study clearly show that the dissolution of C₃S is not affected by superplasticizers but there are strong indications that the nucleation and growth of C-S-H is suppressed.

Experiments were carried out on diluted and stirred C₃S suspensions as well as on C₃S and cement pastes (w/s 0.4). The nucleation and the growth of C-S-H phases were monitored by high resolution scanning electron microscopy (Nova NanoSEM, ESEM XL 30, FEI). The influence of imaging conditions on structural damage of cement hydrate phases is discussed.

INVESTIGATIONS OF THE HYDRATION BEHAVIOUR OF CAC UNDER THE INFLUENCE OF ACETATE AND CHLOROACETATES AT DIFFERENT TEMPERATURES (10°C, 25°C, 45°C & 60°C) USING CLUSTER ANALYSIS

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Summary

The hydration behaviour of calcium aluminate cements can be controlled by using different additives. A combination of additive type and crystallochemical influence was studied by using different concentrations of calciumsalts of acetate $[\text{Ca}(\text{CH}_3\text{COO})_2]$, monochloroacetate $[\text{Ca}(\text{CH}_2\text{ClCOO})_2]$, dichloroacetate $[\text{Ca}(\text{CHCl}_2\text{COO})_2]$ and trichloroacetate $[\text{Ca}(\text{CCl}_3\text{COO})_2]$ at varying temperatures (10°C, 20°C, 45°C and 60°C). For these studies a specially modified isothermal heat-flow calorimeter together with a modified sample preparation technique was used. The additive free cement samples showed a unique setting behaviour with temperature rise from 10°C to 20°C. Calorimetric data of acetate containing samples indicates that hydration is strongly influenced by the parameters temperature, additive and its concentration hence different hydration effects such as light acceleration, strong retardation or even suppression of hydration. Therefore different acting mechanisms can be proposed. Temperature as one of the modified parameters seems to play a very important role. The best retarder in the investigated system was *Calciummonochloroacetate* at every investigated temperature. The development of different hydration products was followed by XRD-measurements. XRD analysis of additive free reference cement samples showed temperature dependent hydration products. Samples treated at low temperatures showed CAH_{10} and C_2AH_8 . Higher temperatures promoted the formation of $\text{C}_2\text{AH}_{7,5}$, C_2ASH_8 and C_3AH_6 . In the additive containing samples besides known phases like CAH_{10} , C_2AH_8 , $\text{C}_2\text{AH}_{7,5}$, C_2ASH_8 and C_3AH_6 also C_4AH_{13} and $\text{C}_4\text{ACcH}_{11}$ as well as **Acetate** containing calcium/aluminium hydrates were formed. Also the formation of acetate bearing gel phases can be proposed. The formation of C_3AH_6 at higher temperatures was only suppressed in the case of *Calciummonochloroacetate*. The use of cluster analysis of X-rays of hydration products is given. Also for the first time cluster analysis on calorimetric data is included.

Keywords: *Acetates, Additives, HAC, Heat-flow Calorimetry, Hydration*

MICROSCOPIC EXAMINATION OF DEGRADATION PROCESSES IN CEMENT-BASED MATERIALS ASSOCIATED WITH THE MARINE ENVIRONMENT.

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ABSTRACT

Concrete and mortar have been used for construction within the marine environment for a long historic period. Structures including harbors, docks, breakwaters, bridge columns and decks, sea walls and oil rigs to name a few have been produced at least since Roman times. The long-term durability of these materials in this environment has thus always been of considerable economic importance. In many circumstances these cement-based materials have performed perfectly adequately over long periods of time. However, in many respects the marine environment should still be considered severe and therefore the selection of appropriate materials for that environmental situation is of the utmost importance.

This paper discusses the main mechanisms involved in seawater-based attack of cementitious materials. It also describes a series of field and laboratory-based observations made on a number of different cement-based materials which variously experienced alkali silica reaction (ASR), the Thaumasite form of sulfate attack (TSA), and decalcification related processes including popcorn calcite deposition (PCD). Using a series of field cases it is shown that small differences in the concrete/mortar composition used, and / or small changes in the environmental situation can lead to severe differences in the degree and types of degradation developed. This paper also attempts to show that it was often a combination of some of the mechanisms working concurrently that induced the most severe degradation. Likewise, in some circumstances it was the absence or reduction of one or another of the processes that significantly reduced or stopped the overall degradation of the material.

THE THAUMASITE FORM OF SULFATE ATTACK (TSA).

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ABSTRACT

Newly discovered and unusual occurrences of the thaumasite form of sulfate attack (TSA) and thaumasite formation (TF) have now been studied extensively. Using both optical and scanning electron microscopy a substantial database of information describing the location, form, composition and characteristics of TSA and TF in a series of different examples both within the UK and abroad has been developed. This paper describes the form typically taken by the TSA/TF within these various localities. Earlier work defined a four-stage degradation process, resulting in the formation of at least three microscopically distinct forms of thaumasite. This paper takes these basic stages, assesses their presence, and makes some revisions to the characteristics of the TSA reaction and its evolution within lower quality cement-derived materials, such as mortars and masonry.

Keywords: Thaumasite form of sulfate attack (TSA), Form of degradation; Thaumasite formation (TF), field examples.

MICROSCOPIC ANALYSIS OF CALCIUM CARBONATE BASED CRACK-FILLING MATERIAL PRODUCED BY BACTERIA IN A NOVEL TYPE OF SELF-HEALING CONCRETE

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ABSTRACT

A novel type of self-healing concrete in which bacteria mediate the production of crack-filling material has been developed in our laboratory. The mechanism of crack-healing is based on concrete matrix-incorporated dormant but viable spores of specific alkali-resistant bacteria which, after activation by crack-ingress water, produce mineral precipitates by conversion of organic precursor compounds. The quality of mineral precipitates produced appeared, however, to be dependent on species of bacteria and type of precursor compound involved.

Environmental scanning electron microscopic (ESEM) analysis revealed that a bacterial isolate originating from soil samples produced robust elongated sphere-shaped 50-100 μm -sized precipitates from the conversion of calcium lactate. In contrast, another isolate originating from an alkaline soda lake produced larger 0.1-1 mm-sized plate-like precipitates from sodium glutamate. Energy dispersive X-ray analysis (EDAX) indicated that the plate-like mineral precipitates were primarily composed of calcium carbonate. Light microscopic analysis of cracked and subsequently water incubated concrete specimens revealed perfect crack-healing of bacteria-based but not of control specimen. We therefore conclude that bacteria-based self-healing concrete represents a durable and particularly sustainable alternative to classical but also to strictly chemically-based self-healing concretes.

INTRODUCTION

Since the last decade efforts are undertaken by several research groups to develop a new type of concrete which features an inbuilt active crack-healing mechanism. The challenge is to apply a mechanism with a potential life time similar to that of the concrete construction and that only becomes active when needed. The rationale behind the idea is that due to crack formation concrete constructions become more and more permeable over time enabling aggressive ingress chemicals to deteriorate the concrete matrix and embedded reinforcement. Connected cracks may even result in leakage of barrier constructions. To delay these deteriorating processes the self-healing mechanism should become activated upon crack formation resulting in the sealing or effective plugging of the cracks. Such a self-healing mechanism could substantially decrease maintenance and repair costs and furthermore extend the service life of concrete constructions. Most types of commonly used concretes do in fact already feature a kind of self-healing mechanism. However, these are a passive and have a rather limited crack-healing capacity. The passive healing activities are related to the amount and type of still non-reacted binder present in the concrete matrix. Particularly high performance or high strength concretes based on mixtures with a low to very low water-to-binder ratio feature a substantial crack-healing capacity. This phenomenon is due to the

MICROSTRUCTURAL ASPECTS OF SBR LATEX-MODIFIED CEMENT PASTE AND MORTAR HIGHLIGHTED BY MEANS OF SEM AND ESEM

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ABSTRACT Styrene-butadiene rubber (SBR) latex modified cement mortars were prepared with varying polymer/cement-ratios (m_p/m_c) and a constant water/cement-ratio of 0.40. The influence of the SBR latex on cement hydration products in cement pastes is investigated using environment scanning electron microscopy (ESEM), the effect of the SBR latex on the polymer-cement hydration products comatrix structures and the interface structures in cement mortar is also studied by scanning electron microscope (SEM). The results show that the SBR latex facilitates the formation and the stability of AFt in cement paste, delays the formation of C-S-H gel and $\text{Ca}(\text{OH})_2$, but up to 3d the structure of the SBR latex-modified cement paste has no significant difference with that of control cement paste. Perfect mesh network polymer films are formed in SBR latex-modified mortars at the m_p/m_c of 8%. The polymer films are formed by the accumulation of polymer particles, and thin polymer films are observed at a hydration time of 2d. With hydration, the formed polymer films are penetrated by gradually growing cement hydration products. As a result, the organic-inorganic interpenetrating structures are formed in SBR latex-modified mortars by two types of successive phases of mesh network polymer films and cement paste. The weak structure at the interfaces of the aggregates and cement paste is modified by the SBR latex effectively.

KEYWORDS SBR latex; polymer-modified cement paste; polymer-modified cement mortar; microstructure; SEM; ESEM