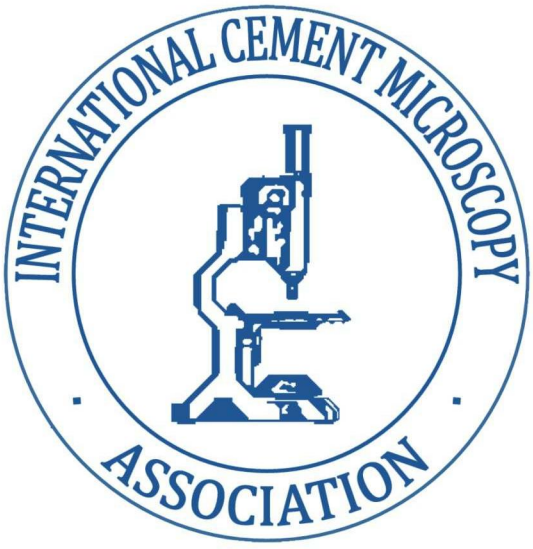
24th INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY



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Alkali Free and Alkali Rich Accelerators for Shotcrete: Physical, Chemical and Mechanical Effects on Cement Hydration

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Abstract

Alkali based accelerators are normally used in shotcrete for tunnels. Such admixtures cause a very rapid set of concrete thus allowing overhead and vertical applications. Today a new class of products starts to be appreciated in the market. They are known as alkali free accelerators. Alkali free and alkali rich flash setting admixtures are based on chemically different compounds which differently affect concrete's properties. A chemical and morphological analysis was carried out on cement pastes mixed with three different setting accelerators (solutions of sodium silicate, sodium aluminate and aluminium sulphate). Setting time, compressive strength and water absorption were also determined.

RHEOLOGICAL BEHAVIOR OF PORTLAND CEMENT PASTES DURING EARLY HYDRATION

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ABSTRACT

This contribution focuses on the structural changes of cement paste from mixing to setting, determined by rheological measurements. Initially after adding of water, the cement starts to hydrate. During the induction period the rate of hydration is very low. The cement paste is still workable and behaves like a fluid. Its viscosity often approximately follows Bingham's law with a yield stress and nearly Newtonian behavior above the yield stress.

During hydration, the structure of the cement paste changes, it stiffens, and both yield stress and viscosity increase. By measuring the rheological properties during cement hydration, information on the structural growth during the hydration process can be obtained. But it is important that the structure of the paste is not influenced by the measurement itself. Cement paste before setting is a flocculated suspension that is very sensitive to the applied shear. Its microstructure breaks down very easily. The usual rheological techniques (for example flow curves) are not suitable for a continuous monitoring of cement hydration, as the yield stress of the paste is exceeded during measurement, causing a structural breakdown.

With a special dynamic measurement technique, low-amplitude oscillatory shear, the structural growth of a cement paste can be studied continuously from mixing until setting. The influence of different admixtures, a retarder and three superplasticizers, was examined.

STRAIN CAPACITY, EXPANSION, DEF, AND ALL THAT!

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ABSTRACT

One of the long-continuing debates in the concrete field has to do with the mechanism of expansion associated with delayed ettringite formation (DEF). The explanations put forward fall loosely into one of two classes: the "paste expansion" theory, in which the driving force is considered to be some form of more or less homogeneous paste expansion induced by deposition of 'submicroscopic' ettringite within the C-S-H, and the "crack expansion" hypothesis, in which the driving force is considered to be the expansive force generated by the growth of ettringite in rim and paste cracks. Expansions produced in laboratory DEF experiments can approach 1.5% - 2%. In this paper we call attention to the facts that (a) the expansion process necessarily involves tensile strain, (b) that hydrated cement paste has a limited tensile strain capacity, usually considered to be about 200 x 10^{-6} (i. e. 0.02%), and (c) that mortars or pastes whose expansions exceed this limiting strain necessarily undergo cracking. Sufficient expansion to induce cracking was shown to take place in most of the mortar bars tested, regardless of whether or not DEF subsequently occurred. For DEF-affected specimens the subsequent expansion, constituting well over 98% of the total strain, took place *after* the specimens had exceeded their tensile strain capacity and were already cracked. SEM illustrations of crack patterns observed in DEF specimens that have just exceeded this limiting strain are provided. It is shown that the subsequent rapid DEF-induced expansions process involves crack widening associated with ettringite deposition. It is difficult to reconcile these findings with any mechanism that postulates that such high expansions can take place while the continuity of the paste is maintained.

ESTIMATION OF THE AMOUNT OF ASR GELS BY THE COMBINATION OF EDS AND PORE SOLUTION ANALYSIS AND MEASUREMENTS OF EXPANSIVE PRESSURE IN MORTARS UNDER RESTRAINT

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ABSTRACT

It is significant for deeper understanding of features of ASR damages of concrete structures to reveal relations between expansive pressure under restraint and free expansions of mortars in the laboratory. One of the purposes of this study is to estimate the amount of ASR gel produced within mortars by the combination of EDS analysis for the gels and pore solution analysis. In addition, this study aims at elucidating relations between expansive pressure measured under a restraint condition, and the amount and composition of gels. The expansive pressure was approximately proportional to the amount of ASR gel formed, when alkali contents of ASR gels formed were less than a critical value. However, mortars containing ASR gels with a higher alkali content than the critical value showed extremely low expansive pressure, even when they greatly expanded in expansion tests without restraint. These results suggest that, in existing ASR affected concrete structures containing gels with a higher alkali content than a critical value, damages due to the secondary stresses caused by restraint might not be so significant, even if reactive aggregates used in the concrete have showed great expansions in mortar bar test in the laboratory.

INVESTIGATION OF FLOOR COVERING ADHERENCE PROBLEM WITH A PORTLAND CEMENT + HEMIHYDRATE SUBSTRATE

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ABSTRACT

An occurrence of adherence problem of a linoleum flooring material with an underlying substrate composed of portland cement and hemihydrate was investigated. The substrate had initially been sealed with an emulsified vinyl acrylic copolymer, and then a water-based linoleum adhesive applied on it after a few months for installation of the linoleum flooring. Apparently, the flooring began to fail prematurely, although a previous laboratory test, using the above procedure and same materials had proven successful.

Petrographic examination of the test sample revealed a reasonably dense portland cement + hemihydrate substrate and its satisfactory bonding with the jute fiber-backed linoleum. The sealer, however, was unidentifiable. In the actual field sample, bonding of the adhesive with the underlying substrate was far from satisfactory; and exhibited a concentration of tiny voids at the upper surface of the substrate. Surface scarification of may have produced increased porosity in the surficial part of the substrate. In fact, this may have contributed to the debonding. The linoleum adhesive, however, has retained its integrity in terms of bonding with the underlying sealer and overlying linoleum. The texture of the adhesive and thickness of adhesive layer were different from the one used in the test sample. Distinct symptoms of debonding at the substrate-sealer interface was observed. Accumulation of a thin layer of a different material was also noted at this interface. Penetration of sealer into the substrate was not observed. The surface of the substrate was carefully scraped from the failed sample and analyzed using Fourier transform infra red (FT-IR) analyzer. Traces of wax was identified in the scrapings. Scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDXA) of the substrate - sealer interface revealed a vacant zone, few micrometers in width, under the sealer, confirming the occurrence of debonding phenomenon at this interface. SEM/EDXA also confirmed the lack of penetration of the sealer in the substrate. The morphology of the crystals directly under the sealer layer was different from those in the interior of the matrix. No evidence of any secondary chemical reaction was noted. .

From differential scanning calorimetry (DSC) of the surface scraping and the interior matrix a preponderance of gypsum ($CaSO_4.2H_2O$) was clearly identified at the surface, whereas the form of calcium sulfate deeper in the matrix was found to be predominantly hemihydrate or $CaSO_4.1/2H_2O$. Thus, a physical conversion of hemihydrate to gypsum had occurred at the surface.

Since the substrate had been left unsealed for quite a few months, it is possible that intake of moisture from the environment over a period of time is responsible for conversion of hemihydrate to gypsum. As formation of gypsum causes volumetric expansion, the sealer applied on this bed of gypsum may have created debondment from the bulk of the hemihydrate matrix. On the other hand, it it is possible that application of wax on the surface of the substrate is responsible for the failure. The wax composed of paraffin, caused the sealer to debond.

IDENTIFICATION OF PHASES IN CEMENT AND CONCRETE USING BACKSCATTERED ELECTRON IMAGING

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ABSTRACT

The backscattering electron coefficient, η, of pure elements was calculated using Arnal's formula. For a number of compounds the average backscattering electron coefficient, $\overline{\eta}$, was then calculated using Castaing's formula. The calculated values showed a linear correlation with measured values of the backscatter electron image intensity in the SEM. The method described can be used to distinguish between phases with similar X-ray spectra, such as calcium hydroxide and calcium carbonate. Furthermore, the porosity of epoxy impregnated phases may be estimated.

EFFICIENCY OF HIGHLY SENSITIVE HEAT FLOW CALORIMETRY IN EXAMINATION OF OPC HYDRATION

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ABSTRACT

The introduced calorimeter is a fully revised, more user friendly equipment based on an arrangement firstly described by Kuzel 1984 [1]. Improvements are: Sample pans made of polystyrene, defined docking of sample vessels to the thermopile, defined sample compaction, and use of a water dispensing aid. The changes of the equipment lead to a very good precision and accuracy of measurements. All measurement were carried out in isothermal mode at a temperature of 23.0 Ä 0.2 °C in a time range of 0 und 72 hours using synthetic clinker like pure phases. All investigations were performed 3 times. Investigations of Alite prove the outstanding precision of the calorimeter with a heat evolution of 205.69 Ä 0.75 J/g (72 h, w/c = 0.45, specific surface 2850 cm^2/g). Kinetics and heat flow are differing with w/c ratio. Belites show a strong dependence of their hydration behavior from the kind of stabilization and modification. α' modification was turned out to be the most reactive in the case of phosphate stabilization. The kinetics of heat evolution from Tricalciumaluminate and Ferrate is depending on their iron content. Ferrate hydration is very fast and reaches 150 – 200 J/g (3100 cm^2/g) within the first hour depending on chemical composition, which is in opposition to Stark (1998) [2] and Odler 1998 [3].

Pathology Observed in Blocks of Concrete Exposed to Marine environment for More than 30 Years old

R. Uribe-Afif, J. Flores-Martinez, L. Franco-Peimbert

Abstract

In the operation maneuvers in a drydock blocks of reinforced concrete are used, these pieces are placed in the low part of the installation and they are used as base as rest of the boats that are constructed or repaired in this area. The use of these elements dates from 30 years old and nowadays it presents signs of a great deterioration. In this work the exposition and service conditions, which it has been exposed during his utility life, are analyzed. The observed pathologies are discussed and identified and is determined the damage level of the elements. Finally recommendations of design for the new structures are made in attention to the prolongation of the durability of the same ones.

1. Introduction

In order to determine the origin of damage of a group of reinforced concrete block's its technical evaluation was made, with this activity is considered to define the deterioration level in the elements, since an important percentage of them has let operate completely in a period of time smaller than 30 years old and those that at the moment continue operating showed evident signs of the occurrence of a pathology. It is important to mention that most of the blocks that most be retired of the operation due at damages level observed in the surface and a condition of disagreeable appearance, and in some cases to great loosening of a great portion of the concrete structure one, fundamentally by blows during its operative handling.

2. Objective

AIR VOID ANALYSIS OF HARDENED CONCRETE WITH A HIGH-RESOLUTION FLATBED SCANNER

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ABSTRACT

A high-resolution flatbed scanner was used to collect an image of a polished cross-section through a concrete pavement. The surface was scanned a total of three times in RGB color. First, the surface was scanned in its natural state prior to the use of stains or treatments. Between the first and second scan, the surface was stained with a phenolphthalein solution to color the cement paste pink. Between the second and third scan, the surface was painted black, and white powder was pressed into the depressions left by air voids. The images collected were aligned, and a new false-color image was created using the B band from the non-stained image, the G band from the phenolphthalein stained image, and the R band from the black-and-white treated image. A principal components transformation was performed on the false-color image to yield a three-band principal component image. The third band of the principal component image and the R band of the black-and-white treated image were input into a classification scheme to yield an output image. In the output image, each pixel is classified as air void, cement paste, or aggregate.

MONOSULFATE, ETTRINGITE, THAUMASITE, OR GYPSUM?

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ABSTRACT

A number of published papers exhibit only scanning electron microscope (SEM) images without X-ray elemental analyses, or X-ray elemental analyses without SEM images, to claim the presence of chemical components containing sulfur in concrete. Results of such "identifications" can be very misleading and may lead to erroneous conclusions about the durability of a particular concrete structure. This paper shows typical BS images accompanied by X-ray elemental analyses of monsulfate, ettringite, thaumasite and gypsum, as observed and identified in concrete.

Key Words: Concrete, SEM, monosulfate, ettringite, thaumasite, gypsum

MICROSTRUCTURAL INVESTIGATION OF SOILS-CONTAMINATED CONCRETE

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and

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ABSTRACT

This paper presents results of microstructural investigation of concrete from sections of slab foundations that exhibited slow hardening characteristic and unusually low strength development pattern. Three samples collected from three different slabs were investigated to determine the cause of the problem. From petrographic examination tiny particles of foreign material were observed to be distributed in fair abundance throughout the paste matrix. Ultra violet spectrum analysis did not indicate the presence of excess superplasticizer. X-ray diffraction analysis, however, showed the presence of clay minerals, which was confirmed from scanning electron microscopy/energy dispersive x-ray analysis. Significant contamination of the concrete with soils, either from the ground on which the slabs were placed or due to contaminated aggregate stockpiles was considered as the most likely cause of the problem related to hardening and strength encountered by these concrete slabs.

THE MINERALOGY OF CHLORIDE ATTACK ON CONCRETE WITH LIMESTONE FILLER

Iver Allan Juel and Duncan Herfort

Aalborg Portland

ABSTRACT

The chloride binding in cement paste prepared from ASTM type II Portland cement and 15 wt % replacement by limestone filler has been investigated. The investigations were performed on small paste specimens at water/binder ratios of 0.7. The specimens were hydrated for 6 months before being immersed for a further 6 months in $CaCl_2$ solutions of different concentrations. This period of exposure was regarded as sufficient for the chlorides to become fully bound by the hydrate phases, and for the solution at this stage to be at equilibrium with the corresponding hydrate phase assemblage. The analytical methods used included XRD and EPMA of the solid phases and standard wet chemical methods for the solution. The results suggest extensive solid solution between Friedel's salt and monocarbonate.

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THE INFLUENCE OF GRINDING AIDS ON THE MICROSTRUCTURE OF CLINKER AND CEMENT HYDRATION PRODUCTS

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ABSTRACT

The grinding aids (GA) are used in order to reduce the mill specific energy consumption. It is commonly accepted that the main effect is to avoid the re-agglomeration of clinker grains in the mill. It is also known that these admixtures can improve the performances of cement.
The aim of our work is to investigate the "mode" of action of alkanolamines based grinding aids. We have focused our attention on morphological modifications of hydration products of clinker and cement.
The microstructures of hydrated products were investigated by ESEM-FEG. The "de-agglomeration" aspect was followed using zeta potential and particle size distribution analyses.
According to our results we can say that during the grinding process "pre-hydration" of clinker occurs in presence of water based solution of GA and that the cement seems to maintain a "memory" of the kind of grinding aids used.

REINFORCING MATERIAL EFFECTS ON HARDENED CEMENT PASTE MICROSTRUCTURES

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ABSTRACT

Cements designed for use in multilateral wells, or in wells where hydraulic fracture is to be performed, are usually subjected to high dynamic loads and high temperature variations. These stresses may cause loss of wellbore zonal isolation if the cement sheath cannot withstand them. Selection of the correct type of cementitious materials, or additives, to reinforce the seal and mechanical properties is very important in order to obtain “a strong” cement that will support mechanical stresses that occur throughout the well productive life.

In this study, mechanical properties and microstructure of reinforced hardened cement pastes containing fibers, latex, and/or resins are evaluated at different curing temperatures and pressure. The effects of adding reinforcing additives as part of the cement pastes in the change of the morphology and microstructure of hydrates formed, as well as the resulting mechanical properties of the cement pastes are discussed.

MICROSTRUCTURAL OBSERVATIONS OF SULFATE ATTACK IN FIELD CONCRETE

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ABSTRACT

Microstructural evolution within a slab of field concrete that has been subjected to sulfate attack is discussed. Associated with the ingress of sulfate from ground water adjacent to the slab, the chemistries associated with sulfate attack alter the microstructure within the slab. Sulfate attack manifests in a variety of ways. These include the formation of ettringite in the paste and at the past-aggregate interface, the development of networks of cracks associated with the ettringite, depletion of $Ca(OH)_2$, elevated porosity which supports transport, and the deposition of other solids containing species from the ground water. The use of a concrete mix design having a w/c ratio near 0.65 imparted sufficient permeability to permit the ingress and internal movement of species present in the local ground water. Species originating in ground water observed in the present study are incorporated in ettringite and gypsum (sulfate), magnesium silicate hydrate (Mg) and Friedel's salt (Cl). The nature of related microstructural alterations and the general locations where they occurred are discussed.

A layer of gypsum had deposited near the bottom of a core extracted from this slab. Calcium also deposited in this region as calcium carbonate. Associated with the gypsum is magnesium silicate hydrate. The formation of this assemblage is the result of combination of carbonation and sulfate attack when the Mg cation is associated with sulfate.

A zone of ettringite occurred at distances further removed from the bottom surface. The ettringite present in this zone formed both in the paste and at the paste-aggregate interface. The porosity in this concrete is not uniformly distributed. Rather, dense and porous regions can be observed in the microstructure. Sulfate attack was associated with the more porous regions. While the normal cementing phases have been completely replaced near the bottom surface of the slab, it is the porous regions which appear to be primarily suffering the effects of sulfate attack in the zones where ettringite has formed. The physical effects of sulfate attack are the depletion of $Ca(OH)_2$ from the interfacial zones and the development of networks of cracks associated with the ettringite zones.

The same sulfate-containing phases were also observed near the top of the slab. Gypsum, infilling both the paste porosity and in fissures in the aggregate, was observed near the top surface of the slab. Below this was a zone of ettringite. Thus, zones of sulfate attack in this slab were propagating upwards from its bottom surface and downwards from its top surface.

Relations and Differences Founded by The Application of ASTM Standards C 295 and 289, The Mexican Experience.

Flores-Martínez, J. J., Montaño-Román, H., Uribe-Afif, R. y Rodríguez-Camacho, R.E.,

Abstract

The evaluation and chemical characterization of aggregates for concrete are supported in the application of independent physical and chemical tests, but because their form of application and objectives that they persecute, can be related. It is the case of those materials that are evaluated to determine their deleterious potential during their use in the manufacture of concrete, in where by his composition and nature, is possible to determine common conditions of origin and/or composition, in such a way that in the process of evaluation of his performance, it can clarify those characteristics that share or distinguish. In the present work, it is analyzed the difference and relation obtained between different aggregates with the application of two different method with a same objective, the method chemical and the petrographic method for the determination of the reactivity potential of the aggregates with the alkalis cement, of which are shown the facts of more than 200 rocks, that correspond in representative form to the more frequently lithologies used in the construction industry in Mexico.

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Scanning Electron Microscopy in Studying Chloride Ingress into Cracked Concrete

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ACI Fellow, **R. Doug Hooton** is a professor of Civil Engineering at the University of Toronto, Canada. He is a member of several ACI committees including 201, Durability; 308, Curing; 232, Fly Ash; 233, Slag; 234, Silica Fume, and 365, Service Life Prediction. He received the Wason medal for materials research in 1988.

ABSTRACT

Although, it is generally recognized that cracks promote the ingress of chlorides in concrete, a lack of sufficient knowledge on this subject does not yet allow reliable quantification of their effects. In the current study, the influence of artificially created, parallel-wall cracks on chloride ingress was examined. Cracked concrete samples were exposed to chloride solution for 40 days. Lateral movement of chlorides from the crack wall into the bulk of the sample was analyzed using SEM/EDX. It was concluded that the lateral diffusion of chlorides was uniform along the crack length. The results of SEM/EDX chloride analysis were in good correlation with results obtained from sample grinding.

A NEW AUTOMATIC ANALYSIS SYSTEM FOR ANALYZING THE AIR VOID SYSTEM IN HARDENED CONCRETE

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ABSTRACT
A new time saving automatic air void analysis system for characterizing the air void system of hardened concrete has been tested on nine previously well-documented concretes.

The sample preparation preceding the automatic analysis involves contrast enhancement steps ensuring white air voids in a black concrete (aggregate and paste). For a well-lapped sample of good quality concrete the contrast enhancement procedure requires 30 minutes to perform. Subsequently, the sample can be analyzed in less than 17 minutes traversing 2413 mm (95 inch) – a vast improvement compared to the several hours normally required to perform linear traverse analysis.

The air void system of the nine concretes investigated was previously analyzed by thirteen European laboratories of which seven laboratories were using a manually method of analysis, and six were using an automatic method of analysis. These nine concretes, representing good as well as poor air void systems, were analyzed multiple times using the new automatic analysis system in order to establish the system's repeatability, as well as to compare the estimated air void system parameters to the other thirteen laboratories.

A very good agreement was found between the air void system parameters measured by the automatic analysis system, and the parameters reported for the thirteen European laboratories. The repeatability expressed in terms of standard deviations of the measured total air contents, specific surfaces, and spacing factors of the new automatic analysis system were found to be as good as the repeatability values provided in ASTM C 457.

Improved Durability Through Nano-Scale Seeding Admixtures in Portland Cement

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ABSTRACT

Previous studies have shown the increased durability in concrete structures in which an alkaline earth silicate admixture was utilized (1). This study was directed at determining the physical properties of the admixture to determine the mode of enhanced properties. Tests were performed on both the admixture and paste specimens that were prepared with and without the admixture. Analytical methods showed the admixture to contain 5 nanometer diameter C-S-H particles within an alkaline solution. The efficacy of seeding as a method of microstructure and phase control has been clearly demonstrated in systems outside of Portland Cement systems. The advantages of seeding and chemical additions to Portland Cement may allow for the simultaneous growth and nucleation of phases for the formation of a durable concrete.

IN SITU OBSERVATION OF HYDRATING CEMENT-CLINKER-PHASES BY MEANS OF CONFOCAL SCANNING MICROSCOPY – FIRST RESULTS

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ABSTRACT

Many previous investigations have been conducted on the hydration of cement.
This work presents a new method for pursuing the hydration of cement with confocal scanning microscope techniques (CSM).
The hydration of pure cement clinker phases were observed after the addition of either calcium sulfate solution or pure water. For the case of the aluminate ($3CaO*Al_2O_3$) hydration in the presence of sulfate two phases of crystallization were observed:

- A first rapid growth of a layer covering aluminate grains with long needle like crystallites
- A second, slower growth of a inner layer existing of smaller crystallites

A new perspective for a better understanding of the crystallization and changes in micro- and mesostructure of cement based materials is given.

MACROSCOPIC TO MICROSCOPIC ANALYSIS OF CONCRETE BY SCANNING ELECTRON MICROSCOPIC MONTAGE

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ABSTRACT

Concrete is a composite material consisting of a binder phase (cement paste), aggregates (rock or mineral phases), and air voids. During external sulfate attack of concrete most of the alteration of the concrete microstructure takes place in the binder phase. However, the alteration is not homogenous throughout the concrete. Some of the local (microscopic) events have profound impact at the macroscopic scale and ultimately on the concrete product. Typical SEM analyses limit the visualization and analysis of microstucture to a single field of view (a few micrometers to a few millimeters) at a time. A collection of images (hard copy or digital) acquired with overlap on all sides can be montaged, but this is a labor-intensive process. When individual, contiguous digital images are acquired under computer control, montaging becomes rather simple. The resulting montage, comprised of perhaps 1000 field images, covers an area of a few square millimeters to a few square centimeters and reveals macroscopic information. Montages consisting of a smaller number of original images (e.g., 5x5) or any of the individual field images provide microscopic information. Including the use of montages in the analysis of concrete has proved to be helpful in linking the information obtained at the microscopic scale where chemical changes are taking place to macroscopic scale where the physical distress is visualized. The usefulness of montaging is illustrated using a case study of a concrete from a residential home.

Keywords: Sulfate attack, montage, SEM, microscopic, macroscopic

METHODS FOR DETERMINING UNKNOWN CEMENT HYDRATION PRODUCTS

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ABSTRACT

This paper presents the results of the examination of cement hydration products recovered from several petroleum wells in Alberta Province, Canada. Cement plugs had been placed in these wells approximately a year earlier, and the operator determined that the plugs had deteriorated and did not have the needed integrity to provide long-term zonal isolation. To determine what could have caused this apparent deterioration, the operator drilled through several of the plugs and submitted the cuttings for examination.

This investigation included ESEM microscopy, X-ray diffraction (XRD), nuclear magnetic resonance (NMR), standard wet-laboratory water analysis, and testing of contaminated slurries.

The relatively cold temperatures under which the cement hydrated, with the presence of the needed chemicals, indicated the possible formation of thaumasite. Thaumasite is a hydration mineral similar to ettringite and could have been the primary cause of cement deterioration in the Alberta Province wells. The formula for thaumasite is $Ca_3Si(CO_3)(SO_4)(OH)_6 \cdot 12(H_2O)$.

Investigation continues, and the authors anticipate that the compound(s) present can be identified.

Mineralogy of modified steel slags

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ABSTRACT

Steel slags are produced during the refining of iron to obtain steel. The transformation occurs by blowing oxygen into the molten pig iron and the addition of lime, limestone or dolomite to entrap the impurities.

During this process, the current practice is to add more lime than the stoechiometry would require in order to improve the efficiency of the refining. This leads to a large amount of free lime in the final slag, with the consequence of expansion when these types of products are used as aggregates or cementitious additions. The solutions which are usually proposed (weathering, carbonation of the slag ..) do not give satisfactory industrial results or have very limited utilisation (addition to the cement raw meal). Two processes, however, have a possible potential to industrial development. Both processes propose a modification to the chemistry and to the mineralogy by an addition treatment (addition of silica) between the converter and the slag discharge process step ('Upstream' process modification).

With this modification, it is possible to eliminate the free lime and to obtain sound aggregates or a product with cementitious properties similar to blast furnace slag. In this study we compare the mineralogy of the final product obtained by both processes and its impact upon the reactivity.

RESUME

Les laitiers d'acièrie sont produits lors de l'affinage de la fonte en acier. La transformation s'effectue par soufflage d'oxygène dans le fer liquide et l'addition de chaux, calcaire ou dolomite pour capter les impuretés.

Pendant le procédé, la pratique courante consiste à rajouter plus de chaux qu'il est nécessaire pour améliorer l'efficacité de l'affinage. Ceci conduit à de grandes quantités de chaux libre dans le laitier final, avec pour conséquence des phénomènes d'expansion quand ces produits sont utilisés comme granulats ou addition au ciment. Les solutions qui sont proposées d'habitude (éventement, carbonatation,...) ne conduisent pas à des résultats satisfaisants industriellement ou à des applications limitées (addition au cru de ciment). Deux procédés semblent trouver une application industrielle. Les deux proposent une modification de la chimie et de la minéralogie par une addition minérale (silice) entre le convertisseur et le parc à scorie (procédé de modification amont).

Avec cette modification, il est possible d'éliminer la chaux libre et d'obtenir un granulat sain ou un produit possédant des qualités hydrauliques analogues à celle d'un laitier de haut fournaux. Dans cette étude, nous comparons la minéralogie des laitiers finaux produits par ces deux types de procédés ainsi que leur réactivité.

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CONTRIBUTION OF SPECTRAL IMAGING FOR CLINKER CHARACTERIZATION

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ABSTRACT

Spectral imaging provides new possibilities for characterization of a material by X-ray microanalysis. This technique consists of acquiring all EDS data at each electronic image pixel. Spectra from any region of the image, X-ray maps and line profiles of any element can be displayed afterwards from the spectral image. Sophisticated software using a recursive pixel allocation algorithm allows sorting of all spectral image pixels by X-ray intensity to allocate pixels to a phase. Then, the system separates "chemically" major phases of the clinker and consequently evaluates phase content with accuracy. The total X-ray spectrum of clinker phases is displayed and then treated by a classical quantification procedure to estimate their chemical composition.

COMPLAINT! ARE YOU SURE IT'S YOUR CEMENT?
Or
Identification of Cement Origin by Oxide Analysis

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ABSTRACT:

Cements, not unlike human fingerprints, are very unique with respect to their chemical analysis. In cases of contamination, it is often possible to look at the major elements of the cement's oxide analysis and determine the origin of the cement. In other cases it might be necessary to compare the major elements oxides with some of the trace element oxides. In cases where the origins are of close proximity, it might be necessary to look at physical characteristics, Bogue Compounds, or infrared scans for organic compounds. It some extreme cases the cements could be so similar that no appreciable difference between cements can be determined, and in these cases contamination between the cements is virtually impossible to determine. In these cases it might be assumed that the cements performance would also be nearly identical and an issue of contamination would never have occurred. This paper outlines the procedures involved in determining the origin of a cement based on its chemical composition. The cements used in this study are all common oil well cements used in the same market, the Gulf of Mexico.

RAPID IDENTIFICATION OF CONTAMINANTS IN CEMENTS

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ABSTRACT

This paper details techniques and equipment that may be used for the rapid identification of various contaminants found in cements over the last several years. Different types of microscopes and related equipment are described and illustrated. Particular emphasis is given to inexpensive microscopes that are easily transported and can be used in field or onsite locations. Results from examining cement samples collected from numerous transportation systems and locations (trucks, railcars, boats, cement plants, and terminals) are given. Methods to help minimize contaminations are also discussed.

ANALYTICAL APPLICATION NOTE

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XRF Analysis of Cement ASTM C-114 Qualification

XRF has long been acknowledged as the accepted technique for analyzing cement, clinker, and all of the associated raw materials in the cement making process. The defining method for the analysis of these materials is the ASTM C-114 Standard Method for Chemical Analysis of Hydraulic Cement [1], which, for the most part is a series of "wet" analysis procedures. However, by conforming to the "Performance Requirements for Rapid Test Methods", alternative methods may meet "Qualification" [2],[3]. Among the many analytical techniques available, X-ray Fluorescence has the unique capabilities of being able to produce the most accurate and precise results, while doing so in the shortest amount of time. Moreover, not all techniques can meet the "Qualification" for all of the elements. In particular, most cement producers consider the analysis of the standard eight oxides, CaO, SiO_2, Fe_2O_3, Al_2O_3, SO_3, MgO, Na_2O and K_2O, to be extremely important to the efficiency of their process. This article describes the ASTM C-114 Qualification method using the Bruker AXS SRS 3400 and S4 EXPLORER, the sample preparation method, the results, and the calculations made to show "Qualification". Both the S4 EXPLORER and the SRS 3400 meet or exceed ASTM C-114 Qualification specifications, while also allowing users to meet more demanding budget requirements.

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Advances in Quantitative XRD Phase Analysis of Cement Clinkers Part I: The Answer to Automation Limits

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