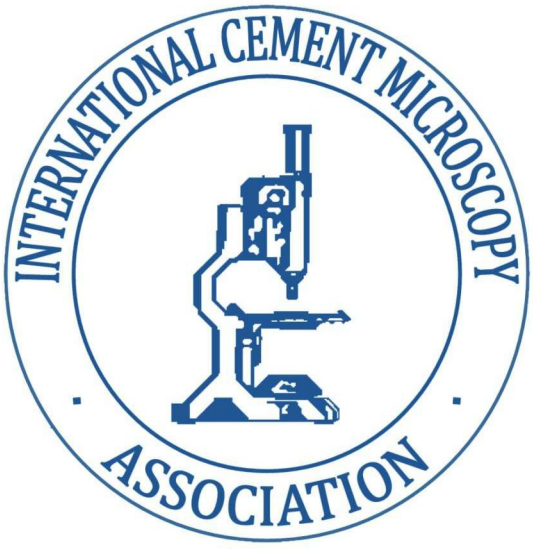
22nd INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY



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Clinker Formation and the Value of Microscopy

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ABSTRACT

Evaluation of clinker using microscopy is a powerful technique that can help improve clinker production and cement quality. One can gather remarkable information about clinker history as well as provide information about cement performance. The key to using the microscope is understanding the process of clinker manufacture: how raw materials are transformed into clinker. The transformation involves both chemical and physical processes, as the material passes through the kiln system.

To produce the desired calcium silicate phases of portland cement, specific raw materials are required. The burnability of the mixture of these materials is determined by chemical composition, mineralogy, and fineness. To convert the raw materials to the cement compounds, the ground feed goes through a series of clinkering reactions in the kiln system. Formation of the primary clinker phases (alite, belite, tricalcium aluminate, ferrite) during this time depends on the pyroprocessing conditions and material properties. At the same time in the kiln, clinker nodules are formed by agglomeration due to capillary forces of the liquid.

The microscope is used to observe features such as clinker phase size, morphology, abundance, and distribution. These observations can not only provide information about the clinker production parameters, but can also offer clues to cement performance properties.

Based on understanding of the process of clinker formation, interpretation of the microstructure can be used for quality control, troubleshooting, and to monitor process changes at the cement plant.

Effect of the fine chemistry of Portland cement clinker on its mineralogical composition

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ABSTRACT
It is possible to determine the mineralogical composition of Portland cement clinker by analytical methods such as points counting, image analysis by mean of optical or scanning electron microscopy (OM or SEM), X ray analysis by Rietveld method, chemical selective dissolution or Bogue calculation. When applied to more complex system i.e. containing minor phases such as alcalies or mixed sulfates (alcalies, earth-alkalies), these methods are not sufficient to predict the total phase composition and further its consequence to the properties of the cement.
In this paper, we use a trial and error method based on the microanalysis by microprobe of the main phases. This method allows a calculation of the percentage of the major phases fitting with the mass balance of the main four oxides in the range of the standard deviation of their chemical analysis. From the residu of the calculation, is then possible to identify the minor elements forming alcalies and earth alkalies sulfate and to estimate their quantity.
It is shown that the effect of these minor elements on the mineralogical composition of the clinker can be decomposed into an indirect effect due to the modification of the burnability which induces a decrease in the alite content (and a simultaneous increase of the belite content) and a direct effect when new phases occur after the saturation of the major phases.

RESUME
Il existe de nombreuses méthodes pour déterminer précisément la composition minéralogique du clinker de ciment Portland, telles que comptage, analyse d'images par microscopie optique ou électronique, diffraction X classique ou par les techniques Rietveld, dissolution chimique sélective, ou calcul de Bogue. Quand elles sont appliquées à des systèmes plus complexes, c'est à dire contenant des phases mineures telles que sulfates alcalins ou sulfates doubles alcalins, alcalino terreux, ces méthodes ne sont pas suffisantes pour prédire la composition minéralogique des clinkers et ses conséquences sur les propriétés des ciments.
Dans ce travail, nous avons utilisé une méthode itérative basée sur la composition chimique des quatre phases principales par microsonde électronique. Cette méthode nous permet de calculer un pourcentage de ces phases qui permet de boucler un bilan massique des quatre oxydes principaux dans la limite de l'écart type de leur analyse chimique. De l'analyse des résidus, il est possible d'identifier les éléments mineurs susceptibles de former les sulfates solubles et d'en estimer leur quantité.
Nous montrons que l'effet des éléments mineurs peut être décomposé en un effet indirect causé par une modification de l'aptitude à la combinaison des matières premières qui provoquera une diminution de la quantité d'alite (et simultanément une augmentation de celle de bélite) et un effet direct lié à l'apparition de nouvelles phases aprés saturation des phases principales en éléments mineurs.

Compositional Analysis of NIST Reference Material Clinker 8486[1]

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Abstract

Certification of the phase compositions of the three NIST Reference Clinkers will be based upon more than one independent method. The current reference values were established using an optical microscope examination, with additional optical microscope data taken from an ASTM C 1356 round robin. The present X-ray powder diffraction (XRD) study provides the second, independent estimate of the phase abundance. Reitveld refinement of the powder diffraction data allowed calculation of a set of best-fit reference patterns and their scale factors. Because of significant contrast in the linear absorption coefficients of ferrite and periclase, relative to the estimated mean matrix linear absorption coefficient, the scale factors were adjusted for microabsorption effects. The XRD data agree with the optical data with the exception of aluminate. This disagreement may reflect the difficulty in resolving this fine-sized phase using the optical microscope. The XRD data did show greater precision than replicate measurements by microscopy.

Measurements from different sources, laboratories, instruments, and from different methods can exhibit significant between-method variability, as well as distinct within-method variances. The data sets were treated using both unweighted and weighted schemes to establish the best-consensus values and to provide meaningful uncertainties. While the mean values of individual phase abundance do not vary, the 95 % uncertainty level values do. The Mandel-Paule-Vangel-Rukhin method of combining the data sets is favored as this method produces a weighted mean whose weighting scheme does not necessarily skew the consensus value in the direction of the large number of XRD values, and that takes between- as well as within-method variation into account.

ALITE VARIATION BETWEEN CLINKERS

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ABSTRACT

The predominant alite polymorphs in each of nine cement clinkers have been determined by XRD analysis. These have then been related to the chemistry of the clinkers and of the constituent phases, in particular the alite composition. It has been found that in these examples, with similar contents of MgO, the SO_3 content of the clinkers was related to that in the alite crystals and that this was in turn closely related to the alite polymorph present, with higher SO_3 contents favouring the inversion to the lower temperature monoclinic form M_1. A works example is presented which suggests that variability of SO_3 due to process factors has affected the type of alite in production clinkers.

SOLID SOLUTION SERIES OF FERRATE AND ALUMINATE PHASES IN OPC:
PART I. THE FERRATE PHASE

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ABSTRACT

X-ray diffraction analysis supported by Rietveld refinement allows quantitative phase analysis simultaneously with determination of the iron content of the Ferrate phase. The difference between actual and calculated chemical composition is determined to be x ± 0.06, related to the common formula $C_6A_xF_{3-x}$. Variable chemical composition, changing distribution of Al^{3+} and Fe^{3+} on tetrahedral and octahedral sites and variable preferred orientation of the Ferrates are prohibiting quantitative XRD analysis using single peak methods. Ferrates from technically produced OPC clinkers which incorporate additionally Mg and Si in their structure can also be analyzed by XRD methods. The chemical composition of this Ferrates is variable with very low iron contents down to $C_6(A,S,M)_{2.39}F_{0.61}$. Changing operating conditions as well as variation of the raw mix influence the chemical composition and the quantitative content of the Ferrate phase.

THINGS PETROGRAPHIC EXAMINATION CAN AND CANNOT DO WITH CONCRETE PART TWO SOME BASICS AND GUIDELINES

By

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ABSTRACT

Our routine procedures for Petrographic Examinations of Hardened Concrete are discussed. A new test for ettringite is proposed. This also discriminates between ettringite and thaumasite.

Examination of Possible Surfactant-Enhanced Ettringite Formation in Hydrating Oilwell Cements

Prepared for presentation at the Twenty-Second International Conference on Cement Microscopy, Montreal, Canada, April 29-May 4, 2000.

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Abstract

This paper presents the results of a study examining the interaction between oilwell cements and different types of surfactants used in petroleum industry spacers/flushes to determine if they enhance the formation of ettringite during cement hydration. Spacers and flushes are used to separate drilling fluids from cement, to help remove drilling fluids, and to leave all contacted surfaces water-wet to enhance bonding with the cement.

This investigation uses scanning electron microscopy (SEM) and quantitative X-ray diffraction to examine ettringite crystal formation in cement samples. Samples of two oilwell cements were tested with and without the following surfactants:

- an alkylarylsulfonic acid
- an ether sulfate
- a water-wetting sugar lipid

All surfactants tested appear to slightly increase ettringite formation in a low-reactivity Class H cement (Cement N), while no appreciable increase was noted in a more typically reactive Class H cement (Cement S). It is therefore assumed that surfactant-enhanced ettringite formation is not a universal phenomenon.

DURABILITY OF RECYCLED GLASS AS AGGREGATE FOR CONCRETE

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ABSTRACT

This contribution deals with the use of recycled glass as aggregate for concrete. The aim of the research work is to make the application of stained glass as colouring component for façade slabs possible. When using glass in concrete, the problem of alkali-silica reaction (ASR) between the alkaline pore solution of the hardened cement paste and glass aggregate has to be considered.
First, investigations were carried out with mortars. In a second series of tests the behaviour of glass in concrete was examined. Different mortar and concrete mixtures using additives and admixtures were investigated to find a mix composition which minimises the effects of ASR.
The damage-causing reactions were measured by the development of compressive strength, dynamic modulus of elasticity and expansion under different storing conditions (storing time up to one year). Microscopical investigations were carried out using scanning electron microscopy and petrographic microscopy on thin sections.

ZUSAMMENFASSUNG

In diesem Beitrag wird die mögliche Verwendung von Recyclingglas als Zuschlag für Sichtbeton untersucht. Ziel ist ein Einsatz des bunten Glases als farbgebende Komponente für Fassadenplatten. Bei der Verwendung von Glas in Beton ist jedoch das Problem der Alkali-Kieselsäure-Reaktion (AKR) zwischen den Alkalien des Zementes und dem Glaszuschlag zu berücksichtigen.
Die Untersuchungen wurden zunächst an Mörteln durchgeführt und in einer zweiten Versuchsreihe auf Betone übertragen. Dabei wurden durch Einsatz von Zusatzstoffen und Zusatzmitteln verschiedene Mörtel- und Beton-Rezepturen untersucht, um eine geeignete Zusammensetzung zu finden, bei der die Auswirkungen der AKR minimiert werden. Die Schadensreaktionen wurden insbesondere mittels Messung der Druckfestigkeit, des dynamischen Elastizitätsmoduls und der Expansion der Prüfkörper unter verschiedenen Lagerungsbedingungen (Lagerungsdauer bis zu einem Jahr) verfolgt. Mikroskopische Untersuchungen erfolgten mittels Rasterelektronenmikroskopie, sowie Lichtmikroskopie an Dünnschliffen.

THREE DECADES OF BRAZILIAN PORTLAND CLINKER MANUFACTURE PROCESS: EVALUATION THROUGH OPTICAL MICROSCOPY

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ABSTRACT

Since optical microscopy was first introduced back in 1969 in the Brazilian Portland Cement Association as a tool to the Portland clinker study, the analysis have been useful to the national cement industry to make constant adjustments to the process by either modernizing the plants or adapting to changes in fuels and raw materials.

Statistical analysis of Portland clinker mineralogical studies carried out from 1970 through 1999 are presented in this paper. Clinker mineral composition and potential quality, manufacture conditions as well as trends expected in the next years are here discussed.

It has been observed that Brazilian Portland clinkers produced in the past decade show distinct characteristics from those produced in the 70´s and 80´s as a consequence of numerous adaptations and improvements in the industry made with the goal to attain an ever better product with optimized costs. Environmental issues are today a major concern of the industry requiring control of the process and product.

CONTRIBUTION OF MICROSCOPY TO THE CHARACTERIZATION OF CONCRETE SURFACES PRESENTING LOCAL TINT DEFECTS

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ABSTRACT

Characterization tests have been carried out on concrete surfaces presenting locally tint defects characterized by alternating light and dark zones. Polarizing optical microscope observations of thin slides associated with backscattered electron examinations (atomic number contrast) and chemical maps made with a scanning electron microscope coupled to energy dispersive X-ray spectrometer, allow to clearly differentiate light zones from dark ones. Results show particularly that iron is not at the origin of the tint of dark zones and that potassium concentration is higher in light zones than in dark zones. Mineralogical constituents contained in both zones are essentially calcite and hydrated calcium silicates. The mineralogical difference between light and dark zones appears essentially quantitative : the light zones contain more calcite and the dark zones more C-S-H hydrates.

RÉSUMÉ

Des caractérisations de surfaces en béton affectées par des défauts de teinte locaux impliquant une alternance de zones de teinte claire et sombre ont été réalisées. Les observations des lames minces au microscope optique polarisant associées aux observations en électrons rétrodiffusés (contraste chimique) et aux cartographies chimiques effectuées au MEB couplé à EDS, permettent de différencier les zones claires des zones sombres. Les résultats montrent entre autres que le fer n'est pas à l'origine de la teinte des zones sombres, que la concentration de potassium est plus forte dans les zones claires que dans les zones sombres. Les assemblages minéralogiques des deux types de zones sont de la calcite et des CSH.

MICROSCOPY A TOOL FOR SUSTAINABILITY

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ABSTRACT

Cement producers are nowadays facing a great challenge to produce, more, environmentally friendly products in order to satisfy their clients and respect their commitment with the environment as well as being competitive in the market.

Ciment Québec is sparing no efforts regarding all of these points. We try to learn from our past and prepare ourselves for the future. The clinker production issue is surely a most important goal.

These targets have been reached, by updating our process, by minimizing wastes, by upgrading our production and continuously improving on the quality of our product as well as working on the development of new products.

Preheater Optimization by Analysis of Decarbonation Degree in Hot Meal

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ABSTRACT

The precalciner outlet material (hot meal) of Ordinary Portland (OP) cement production is an intermediate product mainly resulting from decarbonation and dehydration of the raw material at temperatures up to 900°C. Since the decarbonation reaction is highly endothermic, about 60% of the total required process energy is consumed. For preheater optimization a process dependent decarbonation degree has been established, which can be controlled.

Up to now this degree of decarbonation is determined off-line by CO_2-content or by loss of ignition. In order to ensure effective process control an on-line determination method is required. And additionally it was proofed by our investigations, that there exists no clear correlation between loss of ignition and the degree of decarbonation. For these reasons an alternative direct method using X-ray diffraction supported by Rietveld analysis for quantification of Calcite content in hot meal was worked out.

EXAMINATION OF CLINKERS FROM FOUR SCANDINAVIAN PLANTS WITH RESPECT TO MICROSTRUCTURE AND CEMENT PROPERTIES

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ABSTRACTS

Clinkers of different types produced at four different plants in Scandinavia have been examined with respect to microstructure of clinker and cement properties. The potential compressive strength development until 28 days of each clinker has been predicted from mineral composition and structure. The composition and structure are described by the XRD-profiles in the 2θ-regions 29.88 – 30.70° and 32.90 – 34.10° (using CuK_{α} - radiation). The profiles have been interpreted with respect to mineral structure and composition. Scanning electron microscopy (SEM) of the clinker samples has been performed. The results from XRD and SEM have been related to the typical process conditions in the respective kilns.

The examined clinkers differ in mineral structure and composition and predicted potential compressive strengths at 1, 2, 7 and 28 days. The mineral structure and composition are probably results of chemical and material composition in raw meal as well as production conditions in the kilns. For diagnosing the process condition in the kilns from microstructure in clinker, characterisation of the microstructure by SEM gives more valuable information than characterisation by XRDA does. The SEM characterised microstructure reflects different heating and cooling rates, which is due to the different sizes of the kilns and the different types of cooler. XRDA of cement gives, on the other hand, a satisfactory characterisation of the microstructure of the clinker minerals of the cement with respect to prediction of the compressive strengths of the cements. The predicted compressive strengths of the cements, produced from the examined clinkers, correspond well with the respective measured ones.

SCALING OF CONCRETE DUE TO REVERSIBLE REACTIONS OF SODIUM SULFATE

by

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ABSTRACT

Scaling of surfaces of hardened concrete due to hydration/dehydration of highly soluble sodium sulfate (thenardite/ mirabilite), often a major component of efflorescence in many coastal and desert areas, is frequently undiagnosed or misdiagnosed. It is not "sulfate attack". Sodium sulfate may be found within and below exposed surfaces, or as a thin deposit at a depth of about 3 to 4 mm parallel to the exposed surface. With the exception of cracks near this deposit, the presence of sodium sulfate does not produce distress. The body of the concrete remains sound and does not lose strength. In many cases, a trace amount of sodium sulfate on scaled surfaces can only be identified by using Scanning Electron Microscopy (SEM). Typical SEM images and X-ray elemental analyses of such occurrences are presented.

Key Words: Sulfate attack, reversible formations of sodium sulfate, scaling

PROBLEMS ENCOUNTERED WITH THE USE OF MUNICIPAL SOLID WASTE INCINERATOR BOTTOM ASH IN CONCRETE

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ABSTRACT

The objective of this research was to examine whether municipal solid waste incinerator (MSWI) bottom ash could partially or entirely replace natural coarse aggregate (4/20 mm) in the production of usual concrete (28-day strength of 25 MPa).

The metallic aluminum contained in bottom ash reacted with cement leading to the emission of hydrogen and swelling of concrete. The SEM investigation pointed out that aluminum gels (bayerite and gibbsite), calcium aluminate [$Ca(AlO_2)_2$] and complex silico-aluminate hydrate [Na_2O, 1.06 Al_2O_3, 1.6 SiO_2, 1.6 H_2O] were formed during this reaction. Consequently, the compressive strength of concrete dropped and cracks appeared.

In order to avoid such degradation, a treatment of bottom ash by sodium hydroxide was proposed and allowed a partial replacement (up to 50 %) of coarse aggregate without affecting the durability of concrete.

ETTRINGITE: NOT JUST IN CONCRETE

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ABSTRACT: The easiest way to find ettringite crystals is to break up old concrete and examine the air voids with a hand lens. Outside of concrete, there are few places where ettringite can be found. Natural deposits of ettringite are generally associated with metamorphosed limestones, but have also been reported in modern beach sands (1,2,3,4,5). Under certain conditions, lime-stabilized road bases will produce ettringite (6,7,8,9). A recent examination of a weathered stockpile of blast furnace slag (BFS) on the eastern shore of Lake Superior has revealed the presence of ettringite and gypsum. The dissolution of oldhamite dendrites within the slag is proposed as a source of sulfate for the gypsum and ettringite precipitation.

STATISTICAL DETERMINATION OF CEMENTITIOUS MATERIAL CONTENT IN HARDENED CONCRETE

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ABSTRACT

Determination of cementitious material content in hardened concrete is one of the most important objectives in concrete troubleshooting. Yet it has also been one of the most difficult challenges for concrete technologists for decades. Currently available methods are either too demanding on instrumentation and skills (such as ASTM C1084), or require mix design information that is often not available (such as some methods based on point-count).

An empirical equation has been developed to meet this challenge. It is well known that the paste content of the concrete is related to the cementitious material content and the amount of water added to the concrete. Therefore, the cementitious material content can be determined if the water to cementitious material ratio (w/cm) and paste content are known. The equation was developed based on a statistical investigation of 20 concrete samples with known mix designs covering a cementitious material content from 223 to 505 kg/m^3 (376 to 852 lbs per cubic yard) and w/cm ratio from 0.30 to 0.70. The equation has a coefficient of determination (R^2) of 0.98 and a standard deviation of 11.7 kg/m^3 (19.7 lbs/yd^3). Total cementitious material content can be readily calculated from the equation using the paste content determined by modified point count method and the w/cm ratio determined by traditional petrographic methods. Advantages and limitations of the method were discussed.

KEY WORDS: cementitious material content, regression, point count, w/cm

Evaluation of the structural modification of cement-based systems mixed with latexes

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Abstract

Aim of this study was the evaluation of Portland cement-based systems mixed with different polymer emulsions. In particular we characterised the different samples under physical-mechanical, rheological and morphological point of view.
On this purpose we made 8 different samples mixing the cementitious powder together with 2 polymeric dispersions (an acrylic and a SBR one) at 4 different dosages and constant water/cement ratio.
After curing, the following characteristics were determined on each specimen: morphological (analysis by SEM and Light Microscope), physical-mechanical (viscosity, abrasion resistance by Taber test, crack-bridging test, elongation at break on dumb-bell pieces, water permeability and water absorption). The specific surface areas of these specimens were then determined by means of the helium porosimeter (BET).
We found a very good relation between the physical-mechanical data and the BET ones. By increasing the polymer dosage, we noticed an improvement of the flexibility and of the abrasion resistance of the samples.

DURABILITY OF CONCRETE IN AVERY AGGRESSIVE ENVIRONMENT

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ABSTRACT

In this paper we present the results of a study on concrete deterioration in an aggressive environment. The concrete was used for an evaporation tower construction in the magnesium production industry. The concrete was in contact with brine ($MgCl_2$), hydrochloric acid (low pH) and high temperature (100°C). To achieve this study, chemical, mechanical, and microstructural analyse were performed on concrete.

This study shows that even protected by epoxy paint, the concrete was heavily damaged by the very aggressive environment. Thermal cracking, lixiviation, Ca substitution by Mg in C-S-H and rebars corrosion were the main deterioration processes. Dense and low permeability concrete would decrease significantly the deterioration process.

Chemical Admixture for Sulfate Resistance of Hydrated Oilwell Cements

Prepared for presentation at the Twenty-Second International Conference on Cement Microscopy, Montreal, Canada, April 29-May 4, 2000.

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Abstract

This paper explores the feasibility of using chemical admixture to give hydrated oilwell cements resistance to sulfate attack, and presents preliminary results of tests performed with the admixture. Another objective of the work reported in this paper is to determine whether the same additive can help protect the hydrated cement from drilling-fluid damage. Results observed after 90 days of exposure to sulfates indicate that commonly used oilwell cement, treated with 1% by weight of cement (BWOC) of the admixture resisted sulfate attack as well as the best sulfate-resistant cement available. The untreated, nonresistant ASTM cement bars used in the study have expanded, showing sulfate attack during the 90-day test period.

Light microscopy, environmental scanning electron microscopy (ESEM), expansion bars, and compressive-strength measurements are included in the procedures for studying sulfate and drilling-fluid attack on several hydrated-cement designs. Samples were aged in a 5% sulfate solution and in several drilling fluids at 39°C, and were removed periodically for measurement and evaluation. The paper reports findings of the study and shows (1) photomicrographs of samples and (2) physical measurements recorded.

THERMAL TREATMENT OF RECYCLED CONCRETE AGGREGATE FOR GENERAL USE IN CONCRETE

A preliminary laboratory study

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ABSTRACT

In this paper, the results of a preliminary laboratory study to assess the effectiveness of thermally treating recycled concrete aggregate for general use in concrete are presented. The samples used for the study consisted of sieved fractions of crushed concrete that were subjected to various thermal treatments at temperatures of either 650 °C or 800 °C. In each case, the treatment lasted for a period of 0.5 or 1 hour. After the thermal treatments, the samples were first investigated by means of polarising and fluorescence microscopy followed by strength tests on concrete specimens prepared with one of the treated samples. The thermal treatments caused considerable reduction in the amount of cement paste or mortar adhering to both the fine and the coarse aggregate particles. The reduction was more pronounced in the case of the samples treated at a temperature of 800 °C than those treated at 650 °C. The integrity of the aggregate particles was found to a large extent to be preserved. The strength development of concrete specimens, prepared with one of the treated samples was slower than that of conventional Dutch river-dredged aggregate but reasonably good. Although this is a preliminary study, the results indicate that thermal treatment of recycled concrete aggregate at a temperature of about 800 °C can yield good quality aggregates, with properties which are reasonably comparable to conventionally used Dutch river-dredged aggregates.

A NEW METHOD OF AIR VOID ANALYSIS FOR STRUCTURAL CONCRETE

by

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ABSTRACT

This paper describes the application of high resolution industrial CT x-ray scans to measure air void parameters in concrete in three dimensions. This patent pending technique uses small diameter samples of hardened or fresh concrete that can be scanned at resolutions of up to 10 microns. The resulting dataset is a three dimensional representation of the sample showing the spatial distribution of aggregate, paste and air voids. Software is currently under development to automatically measure specific attributes of the air voids such as air void size and size distribution, mean spacing between voids and surface area. This method has the potential to offer significant advantages over current linear traverse and point count methods due to increased reliability, reproducibility and statistical accuracy. Future work will focus on establishing a more direct link between analytical results and concrete performance.

FREEZING AND THAWING RESISTANCE OF DRY COMPACTED SEGMENTAL RETAINING WALL UNITS

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ABSTRACT

In 1998 the Minnesota Department of Transportation (Mn/DOT) adopted an aggressive testing program, test method and standard for performance of dry compacted segmental retaining wall units. These units are traditionally used in the construction of retaining walls and other similar highway structures. They are produced by vibrating the concrete internally and externally.

The testing program presented consisted of measuring physical properties of the units including compressive strength, density and absorption, as well as running the MN/DOT procedure. In addition, thin sections were prepared from each sample and observed to evaluate the microstructural impact on durability. Woods Metal Intrusion Porosimetry (WMIP) was used to qualitatively review the distribution of pore structure within the paste.

The compressive strength, absorption, and density are relatively poor indicators of performance of the units in freezing and thawing, however there is no clear theory as to why this should be the case. The thin section analysis indicated that those units showing good freeze/thaw performance had been well cured, based upon the relative abundance of hydrated Portland cement. In contrast, those samples which had not shown good performance showed a large amount of unhydrated Portland cement and mineral admixtures in particular in the finer fraction. WMIP showed that the resulting pore structure of the latter units compromised their freeze-thaw durability, in that better cured samples had considerably finer pore size distribution. A model of the microstructure of dry cast retaining wall units is proposed which explains the poor relationship between compressive strength, absorption density and freeze thaw resistance.

PHASE COMPOSITION AND MICROSTRUCTURE CHANGES DUE TO HEATING AND REHYDRATION OF SOME HARDENED BLENDED CEMENT PASTES

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Abstract

The effect of heating and rehydration on phase composition and microstructure of some hardened blended cement pastes as well as the plain cement pastes was investigated. The blending materials used in this investigation were slag, thermally treated kaolin and silica fume. On heating at 200°C, the area enclosed by the broad peak characteristic of CSH increased, but those characteristic of C_3A. CS. H_{12} and C_4AH_{13} were disappeared. Whileas, heating at 600 °C caused a decomposition of all the hydrated phases. On rehydration, of the samples heated at 600 °C, peaks characteristic of CSH were observed, also those of CH but with different intensities. Hardened plain Portland cement paste and that blended with slag showed microcrack formation by heating at 200 °C. Whereas, the pastes blended with thermally treated kaolin or silica fume did not show crack formation by heating at 200 °C. Heating at 600 °C caused a formation of cracks in all samples but with different extents.

MICROSTRUCTURE OF C-S-H, TOBERMORITE AND XONOTLITE SYNTHESIZED IN THE PRESENCE OF SOME ADMIXTURES

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ABSTRACT

The microstructure of hydrated calcium silicates formed under the saturated water vapour at 180ºC was observed by SEM. The lime-quartz mixtures with CaO to SiO_2 molar ratio of 1.00 and 0.83 were prepared and cured hydrothermally for 8, 24 and 72 hours. The effect of some admixtures, such as $CaSO_4$, $CaCO_3$, $Al(OH)_3$, and metakaolinite ($2Al_2O_3 \cdot 4SiO_2$) was investigated. At CaO/SiO_2 = 0.83 the transformation of C-S-H to tobermorite was generally observed. At CaO/SiO_2 = 1.00 the C-S-H phase and tobermorite formed within 8 hours curing; at prolonged curing the xonotlite appeared. The crystallisation of xonotlite was generally inhibited by the presence of Al containing admixtures and perhaps by use of quartz. In many samples the simultaneous occurring of different morphologies as well as the transitions between the particular forms were observed.

PETROGRAPHIC EXAMINATION OF A 67 YEAR OLD CONCRETE BRIDGE DECK

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ABSTRACT

A 67-year-old concrete bridge (J857) on State Highway 72 in the Missouri, USA, has been decommissioned because of realignment of the highway. The 18-inch thick, single reinforced bridge deck bridge was cast in 1932. According to maintenance records, de-icing salts were used for the past 40 years, and an asphalt overlay was placed on the deck in the early 1990's. Although the bridge is very old, the concrete appears to be sound.

Microscopical features observed in cores from the bridge show different characteristics than of concrete used today.

Petrographic examinations of two cores were performed to estimate the mixture proportions of the concrete and compare them to the intended mix design available. Also discussed are characteristics of the concrete constituents. Relict Portland cement particles are present and are characterized. Although the concrete bridge deck is 67 years old, the concrete shows no deep distress.

EFFECTS OF LIMESTONE ADDITION ON MICROSTRUCTURE AND HYDRATION OF CEMENTS

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ABSTRACT

In recent years, with the advent of new cement standards to Europe, usage of limestone as an additive in cement becomes popular. Although the effects of limestone powder on concrete has been studied extensively, the effects of limestone on cement hydration is not investigated thoroughly. Aim of ongoing research is to evaluate the influences of limestone on grindability, physical and mechanical performance, porosity and hydration of cements. The paper presented here gives the results of microstructural investigations on the hardened cement pastes with limestone and the discussions on these. In the study, portland limestone cements with 5 to 30 % limestone were produced by intergrinding the raw materials to 3600 ± 75cm^2/g Blaine fineness. The hardened cement pastes produced by using these cements were investigated at 2 and 28 days. For assessment of the effects of limestone on cement hydration, interactions between calcite and cement paste matrix were investigated. Results yielded that the reaction products tend to accumulate around the limestone particles and CH in the cements with have smaller size than CH of portland cement. Also $CaCO_3$ takes part in hydration reactions of cement.

ÖZET

Son yıllarda yeni Avrupa Standardları'nın kabul edilmesiyle beraber, kalkerin çimentoda minör ya da majör ilave katkı maddesi olarak kullanımı artmıştır. Kalker tozunun, betona etkileri birçok araştırmacı tarafından çalışılmış olmakla birlikte, çimento hidratasyonuna etkisi tam olarak incelenmemiştir. Buradan hareketle, devam etmekte olan araştırmada kalkerin çimentoda öğütülebilirliğe, fiziksel özelliklere, mekanik özelliklere, gözenekliliğe ve hidratasyona etkileri incelenmektedir. Burada sunulan makale, bu araştırmanın hidratasyon mikroyapısıyla ilgili sonuçlarını ve bu sonuçlara dair yorumları sunmaktadır. Çalışmada % 5'ten 30'a kadar kalker katkılı 3600±75 cm^2/gr Blaine inceliğinde portland kalkerli çimentolar beraber öğütme tekniğiyle üretilmiştir. Bu çimentolarla üretilen sertleşmiş çimento pastaları 2 ve 28 günlerde SEM ile incelenmiştir. Kalkerin çimento hidratasyonuna etkisini belirlemek için kalsit ile matriks arasındaki etkileşimler incelenmiştir. Elde edilen sonuçlara göre kalkerli çimentolarda oluşan CH yapıları daha küçük boyutlu ve dağınıktır. Ayrıca reaksiyon ürünleri kalker tanecikleri etrafında toplanma eğilimi göstermektedir. $CaCO_3$ hidratasyon reaksiyonlarına kısmen girmektedir.

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THE INFLUENCE OF TWO-STAGE MIXING TECHNIQUE ON THE STRENGTH AND WORKABILITY OF CONCRETE CONTAINING PFA AND MICROSILICA

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ABSTRACT

This paper presents the results of an investigation into the influence of differing levels of PFA replacement on the mechanical properties of concrete mixes in terms of strength and workability. These mixes have been established by Elkem Materials, Ltd with Tarmac Topmix, UK as basic high quality concrete mixes for road pavement. The mix design contains cementitious material, 10% dosage microsilica and diferent levels of replacement material. The replacement material investigated in this study is pulverised fuel ash (PFA). Conventional and two-stage mixing techniques are used to prepare the tested samples. The microstructures of the material produced is investigated using Scanning Electron Microscopy (SEM, in order to obtain an understanding of the effects of microsilica, PFA and the two-stage mixing technique on the physical properties of the concrete. The concrete mixes with different proportions of cement replaced by PFA are shown to have a high quality in terms of strength, workability and durability. The concrete produced would be an economically attractive material to use as its cost would be greatly reduced.

SEM INVESTIGATION OF MICROBIAL CALCITE PRECIPITATION IN CEMENT

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ABSTRACT

This paper describes the scanning electron microscopy (SEM) investigation on the microbial plugging of artificially cracked cement mortar cubes. *Bacillus pasteurii* ATCC 11859 mixed in sand was used as the filler material. An artificial cut was provided on cement mortar cubes to simulate a crack, the filler material and bacteria were filled into it and the whole specimen was cured in Urea-$CaCl_2$ medium. The filler material at various parts within the crack was investigated using SEM. Distinct crystals were observed on the surface of the crack. It was found that these crystals bound the filler material to the crack and also the filler particles with each other. Energy-dispersive X-ray spectra of the crystals showed abundance of calcium, indicating that the precipitated material was calcite ($CaCO_3$). Furthermore, many calcite crystal faces showed rod-like impressions. On the basis of size and shape, it was inferred that these were the space housed by *B. pasteurii*. These microscopic observations serve to confirm the mechanism of microbial calcite precipitation. It was also possible to study the difference in the precipitation of calcite with the increase in the depth of crack. The SEM was thus found to be a vital tool to establish the behavior of microbial calcite precipitation.

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Effect of Fly Ash on the Microstructure and Performance of Concrete as Studied by AC Impedance Spectroscopy

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Abstract

Nyquist format of AC Impedance Spectroscopy (ACIS) of concrete will show the microstructure and permeability of the specimen. Usually, at high frequency range a semicircle in Nyquist plot will show the porosity and the dimension of fractals of the specimen while at low frequency range a straightline of slope 1 will describe the diffusivity and permeability of it. Fly ash as blend will change the Nyquist plot with the result that one can see how it influences the microstructure of concrete and hence its performance. Furthermore, the mechanism of the change can be elucidated and its dynamics can be estimated.

Application of Cryo-transfer scanning electron microscopy for investigation of cement hydration and cementitious systems

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Abstract

Low temperature stages for scanning electron microscopy (SEM) were developed initially to avoid water loss from hydrated specimen when they are transformed to high vacuum conditions in the electron microscopes. The preparation and shock freezing can be done within some minutes. Therefore it is possible by using this technique to investigate the water containing phases of hydration at very early stages of reaction. Several different applications will be shown. The use of the technique for investigation of hydration of OPC´s, High alumina cements, and the influence of additives will be shown at very early hydration stages. Also the handling, preparation and use of this technique is explained in detail. The development of phases and distribution of phases can be demonstrated by using fractured frozen samples.
Generally the main advantage of this technique is that it is possible to see the phases (crystalline and non-crystalline) in contact with the frozen hydration solution and also the internal structure.

Introduction

Scanning electron microscopy is a long used well known technique in the study of cementitious materials. For hydration reactions and for the study of hydrates we normally face the problem that extreme conditions like high vacuum, electron beam heat etc. influence the phases or even make it impossible to study and analyze the hydrates. The introduction of environmental scanning electron microscopically (ESEM) techniques allows the examination of humid samples and gives the chance to see hydrating cement in a real gaseous world. Even crystallization processes can be made visible out of water containing solutions. Water containing samples and hydrates often show dehydration cracks when normal SEM imaging is used. Therefore the increased focus on other techniques is quite important in cement chemistry. Several authors describe the use of ESEM in studying the hydration process of cement samples and cement hydrates (Caveny,C., Radonjic,M. et al., Damidot,F. & Sorrentino,F., Mehta,S. & Jones,R.).
Another technique the Cryo-transfer microscopy was mainly used for the analysis of biological samples, suspensions, medical problems and nutrition analysis. The application of Cryo-transfer microscopy for the analysis of different types of cements and cementitious materials will be shown in this paper. Also dynamically reactions like the early time hydration of cements will be investigated by cuts at different times. The principle that the samples will be shock dried in liquid nitrogen and be kept cool during the investigation causes that the vapor pressure of water or ice is lower than that of the microscope vacuum. Therefore all the

A STUDY OF PORTLAND CEMENT - GROUND GRANULATED BLAST FURNACE SLAG PASTES SUBJECTED TO LOW PRESSURE STEAM CURING

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ABSTRACT

The present study uses x-ray diffraction and optical microscopy to qualitatively evaluate mineral phases present during hydration of neat cement pastes both with and without ground granulated blast-furnace slag (GGBFS). The present study identifies the products of hydration in pastes made with Type IIIB Portland cement and GGBFS exposed to varying curing regimes including a low pressure steam cure. Comment is made regarding effects that the hydration of the Type IIIB Portland Cement/GGBFS and various curing regimes have on the potential for development of efflorescence as compared to mixtures of neat Type IIIB Portland cement pastes.

The quantities of calcium hydroxide in the Type IIIB/GGBFS pastes are lower than ordinary Portland cement pastes throughout all ages. Early age reduction is the result of the reaction of the glass phase in GGBFS with calcium hydroxide, as well as, the dilution of lime-containing Portland cement compounds. Further reduction of calcium hydroxide due to hydration reactions of the calcium hydroxide with the glass phase of the GGBFS continues to occur after 365 days. The hydration products at later ages of the pastes cured initially in low pressure steam conditions and stored in 100% relative humidity were virtually identical to those pastes of similar composition cured initially at ambient temperature and stored in 100% relative humidity.

ESEM STUDY OF THE HYDRATION OF THE SILICATE, ALUMINATE AND FERITE SINGLE PHASES FROM PORTLAND CEMENT

by

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ABSTRACT

The major functional properties of concrete and mortar depend on the hydration and subsequent carbonation of cementitious components used as a binder. In order to clarify the reactions of each major constituent of Portland cement, 'in vivo' observations in a humid environment were necessary requiring Environmental Scanning Electron Microscopy (ESEM) techniques.
Samples of alite, belite aluminate and ferite were examined in powder form; initially dry and then at various degrees of controlled humidity exposure for up to 28 days. The evolution of microstructure in each sample during the hydration process is shown in a series of micrographs. The aim of this paper is to promote and encourage the use of ESEM in the analysis of building materials by illustrating the evolution of various phases during the hydration of alite, belite aluminate and ferite from OPC.

COMPARATIVE STUDY OF CLINKERS BURNED WITH UP TO THIRTY PERCENT SUBSTITUTION OF FUEL FOR WASTE FUEL

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ABSTRACT

A comparative study has been carried out on clinkers burned with up to 30% substitution of fuel for a blended waste fuel. The study aims to check the influence of burning waste on the manufacture process through the evaluation of clinker microstructure as well as to observe the increase on heavy metals in clinkers burned with waste. Cement performances have also been evaluated and leaching tests have been carried out.

Cadmium, Hg, Tl, As, Co, Ni, Se, Ag, Ba, Cr, Cu, Pb, Sb, Zn, Mn, V and Sn contents were obtained by atomic absorption spectrometry; and clinker microstructure analysis, by reflected-light microscopy.

The results show that substitutions for waste fuel have not brought out any visible changes in clinker microscopic features or adverse effects on cements mechanical performances. Heavy metals contents have not increased significantly as to influence clinker properties.

KNOWING CLINKER MICROSTRUCTURE - A POSSIBILITY TO INFLUENCE GRINDABILITY THROUGH TECHNOLOGY

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ABSTRACT

Based on the complex study - chemical-mineral composition, macro and microstructure, grindability -, of about 200 commercial clinkers it was shown that the *grindability - easy, medium and heavy* grindability - *of a clinker* with a given mineral composition is influenced to a major extent by the *microstructure* of the clinker. The microstructure of clinkers is, however, also influenced by production parameters, mainly by *raw meal grinding fineness and homogeneity,* and by *clinker burning and cooling intensity*. The microstructure and microhardness of each clinker phase - and ultimately clinker grindability - is influenced also by the type of fuel used, the trace elements incorporated into the crystal structure, etc. By knowing these relationships it will be possible *to influence clinker grindability and thus the energy used for grinding*.