30th INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY

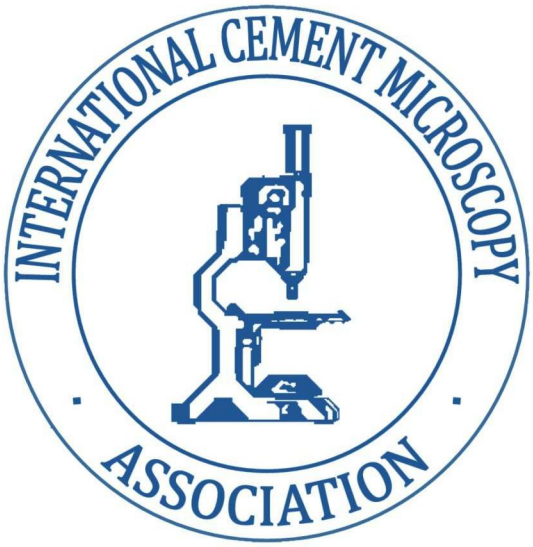

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Table of contents

	Pastes Containing Silica Fume and Admixture
H. El-Didamony	Fire Resistance of Silica Fume – Fly Ash Composite Cement Pastes
B.A. El-Sabbagh, A.M. Kandeel, & T.M. El-Sokary	The Uses of Crushed Potteries Waste for Decorative Pottery Tile Manufacture
D. Jana	DEF and ASR in Concrete – A Systematic Approach From Petrography
P. Stutzman & G. Lespinesse	Cementitious Materials Crystal Structure Database for X-Ray Powder Diffraction Analyses

SUPPLEMENTAL CEMENTITIOUS MATERIALS (SCM) FOR USE IN OILWELL SLURRIES

Tom Dealy, Chris Gordon, Keith Pewitt; Halliburton
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Abstract

As the world becomes more and more environmentally conscious, the role of cement manufacturers becomes ever more critical. The manufacture of Portland cement is a very energy-intensive industry. Globally, cement manufacturers account for 2% of total energy consumption and 5% of total industrial energy consumption. Much of this energy is produced by burning fossil fuels such as coal, and the burning of fossil fuels contributes greatly to the production of greenhouse gases. About 90-95% of the energy used is consumed by the kilns, while 5-10% is used as electricity for lighting, finish mills, and other equipment.

In addition to this, the calcining process that occurs in the kiln releases carbon dioxide (CO_2), another greenhouse gas. It is said that currently cement manufacturing accounts for 5% of the global production of CO_2. Half of that is from energy consumption and half from calcinations.

This paper examines oilwell cement slurries prepared with Portland cement that has been produced utilizing the addition of up to 40% SCM, to determine if they are acceptable replacements for currently used slurries. By substituting SCM for a portion of the cement, a plant can greatly reduce its CO_2 production. Many pozzolanic materials such as flyash and ground blast furnace slag (GBFS) are used as SCM, but many of them must be transported to the cement plant. This incurs freight costs, but perhaps more importantly it requires the burning of additional fossil fuels in trucks, trains, etc. The SCM used in this testing is crushed limestone, which is already present at the plant.

UNDERSTANDING EARLY STAGE CEMENT HYDRATION AT 400°F IN THE PRESENCE OF INORGANIC HOLLOW BEADS—AN SEM STUDY

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ABSTRACT

Increased global demand for energy is forcing oil and gas industry operators to drill and produce in increasingly challenging environments. Pumping cement slurries through highly depleted zones and weaker formations requires slurry density to be lower. However, long-term zonal isolation demands mechanical stability of the cement sheath, even at high-pressure high-temperature (HPHT) downhole conditions.

Inability to successfully circulate the cement and cement sheath failure shortly after placement can have a great impact on the drilling/completion cost and cause delays in production due to the required remedial work. Density of the cement slurries is often lowered by foaming, using hollow inorganic beads, or both. Extremely low-density inorganic hollow beads, made with a high percentage of silica, enable lowering the overall slurry density without much increase in the porosity/permeability of the set cement sheath.

It is well established that prevention of mechanical-strength retrogression under HPHT conditions requires a higher Si/Ca ratio in the slurry design. The ratio of Si is usually increased by adding an additional source of relatively pure silica of some form such as fly ash. What is not as well understood is whether the inorganic beads used to lower the slurry density will be consumed as a source of silica during high-temperature crystallization reaction. This may result in (1) an increase in porosity/permeability and (2) loss of mechanical integrity of the set cement sheath leading to inadequate zonal isolation. In this work, ESEM has been used extensively to monitor the high-temperature cement hydration chemistry at early stages and provide chemical insight into designing a stable low-density cement slurry containing hollow beads that can provide long-term zonal isolation under HPHT conditions (>250°F).

CONCRETE DETERIORATION FROM PYRITE STAINING, SEWER GASES, AND CHIMNEY FLUE GASES – CASE STUDIES SHOWING MICROSTRUCTURAL SIMILARITIES

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ABSTRACT

Case studies are presented to show chemical deterioration of concrete exposed to three different moist, oxidizing environments, where oxidation generated sulfuric acid, which, in turn, has caused acidic corrosion and associated sulfate attacks in concrete. Concrete containing unsound pyrite inclusions in the near-surface aggregate particles commonly show staining and popout, and associated sulfuric acid-induced decomposition of paste around the unsound particles. Concrete liners in chimney environments often show acidic corrosion of the inner wall of liner, carbonation, and white calcium sulfate deposits, from interactions of moist sulfur and carbon dioxide flue gases with the portland cement hydration products. Concrete sewer channels show similar acidic corrosion of the inner wall, mostly in the aerated portion, due to oxidation of sewer gases (hydrogen sulfide) by aerobic bacteria that colonizes on the moist aerated portions. Although the attacks in present cases are primarily of sulfuric acids, the products of such attacks, i.e., gypsum, can cause concomitant sulfate attacks, with microcracking and further decomposition of paste. Corrosion, microcracking, staining, discoloration, leaching and decomposition of portland cement hydration products, carbonation, and gypsum formation in cracks and voids are some common microstructural features that develop from these combined acidic and sulfate attacks, which are similar to the ones observed in many classical external sulfate attacks in concrete from exposure to a sulfate-rich soil. Case studies show the importance of detailed petrographic examinations in evaluating condition of concrete in such environments, depths of attacks, and anticipated future durability.

COMBINATION OF CRYO-SEM, IN-SITU XRD AND HEAT-FLOW CALORIMETRY FOR EARLY TIME HYDRATION STUDIES OF PORTLAND CEMENTS

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ABSTRACT

Advanced cryo preparation techniques open new ways for SEM investigations on hydrating cementitious systems. In this paper the hydration of two different Portland cements is studied with improved Cryo-SEM methods in combination with heat flow calorimetry and in-situ X-ray diffraction. The cements show significant differences concerning their hydration behaviour and their phase development. Cryo-SEM images give essential information on microstructure and morphology of hydration products. Portlandite, AFt- and AFm-Phases are detected by in-situ XRD and Cryo-SEM. The results of the different methods are compared to obtain detailed information about crystallisation processes and microstructural development during the first 3 days of OPC hydration.

Unexpected Severe Deterioration of New Cast-In-Place Concrete Piles - A Case Study

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ABSTRACT

During construction of a multi-story building in a sub-tropical region, underground concrete piles that exhibited severe cracking and spalling around their perimeters were discovered. The exterior surface of the most deteriorated concrete was unusually soft and powdery. Based on petrographic and SEM studies, the concrete deterioration was determined to be caused by massive ettringite development, mainly due to reaction of the cement paste with the surrounding soil and backfill environment and also to the chemical composition of the underground water.

Key Words: Sulfate attack, concrete piles

SWIMMING POOL PLASTER DETERIORATION – OVERVIEW & CASE STUDIES

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ABSTRACT

Deterioration of swimming pool's cementitious coating (plaster) is a complex process involving interactions of four closely related systems – (a) the pool water containing water-treatment and other chemicals, (b) the plaster, (c) the shotcrete or concrete substrate on which the plaster is applied, and, (d) the environments in which the plaster was placed and is in service. Common forms of pool plaster deterioration include: (a) cracking of pool plaster by various mechanisms, such as shrinkage prior to water immersion, structural movement of pool such as settlement of substrate, or, expansive chemical reactions within the substrate putting the plaster in tension; (b) softening of plaster due to the use of inferior plaster materials or mix, improper placement temperature and practices, and interactions with aggressive pool water; (c) debonding of plaster from substrate, or debonding of one coat of plaster from its undercoat; (d) sporadic mottled or blotchy gray-hued abnormal discoloration of plaster surface; (e) staining discoloration from various sources; (f) dissolution of plaster (leaching) and associated etching by aggressive acid water, (g) scaling or precipitation of secondary deposits on plaster surface by alkaline water chemistry; (h) combined aggressive chemical attacks by alternating acidic and basic pool water chemistries on the plaster surface and associated etching and staining; (i) spot alterations from aggressive water attack and/or improper plastering processes; (j) plaster surface popout and local staining by unsound or reactive sand particles in plaster; and (k) freezing and thawing damage of plaster surface prior to water immersion, or of portions not immersed in water, etc.

Petrographic examinations (optical and scanning electron microscopy) and chemical analyses of plaster samples from damaged pools are often helpful to diagnose the cause(s) of plaster deterioration and its extent. Case studies presented herein are of samples received to our laboratory from various pools at different degrees and types of deterioration. Studies discussed include: (a) cracking of pool plasters by excessive shrinkage of plaster and deleterious alkali-silica reaction of aggregates in the concrete substrate, (b) plaster debonding and blistering, (c) plaster etching and scaling by imbalanced pool water, (d) mottled discoloration of plaster, (e) staining by brick masonry copings, and (f) spot alterations and associated spot staining of plaster.

INVESTIGATIONS ON THE CRYSTAL CHEMISTRY OF OXYGEN DEFICIENT PEROVSKITES CRYSTALLIZED IN MANGANESE MODIFIED CALCIUM ALUMINATE CEMENTS (CAC).

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ABSTRACT

Brownmillerite solid solutions with the chemical compositions $Ca_2(Fe_{0.5}Mn_{0.5})_{2-x}Al_xO_{5-d}$, $0 \leq x \leq 4/3$ $x = ⅙$ were synthesized and investigated at the BENSC Hahn-Meitner Institut Berlin Instrument E9. The incorporation of Mn in the structure stabilize the alternate L-R-orientation of TO4 polyhedra. Mn^{3+} occupies exclusively the octahedrally coordinated position 4a (0,0,0) / (x,0,0) I2mb. Increased Mn^{3+} - concentrations cause a remarkable distortion of the octahedron and the tetrahedron indirectly, resulting in twisted octahedral layers and buckled and tetrahedral chains.

The hydration behaviour of CAC under the influence of acetate and chloroacetates at different temperatures (10°C, 25°C, 45°C & 60°C)

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Summary

The hydration behaviour of calcium aluminate cements can be controlled by using additives. A combination of additive type and crystallochemical influence was studied by using different concentrations of calciumsalts of acetate [$Ca(CH_3COO)_2$], monochloroacetate [$Ca(CH_2ClCOO)_2$], dichloroacetate [$Ca(CHCl_2COO)_2$] and trichloroacetate [$Ca(CCl_3COO)_2$] at varying temperatures (10°C, 20°C, 45°C and 60°C). In the additive free cement samples a unique setting behaviour was observed with temperature rise from 10°C to 20°C. The calorimetric results of acetate containing samples indicate that hydration is strongly influenced by the parameters temperature, additive and its concentration hence different hydration effects occurred as there was some slight acceleration and strong retardation or even suppression of hydration. Because of that different acting mechanisms can be proposed. Temperature as one of the modified parameters seems to play a very important role. The best retarder in this system was the **Calciummonochloroacetate** at every applied 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 $C_2AH_{7,5}$, C_2ASH_8 and C_3AH_6. In the additive containing samples besides known phases like CAH_{10} ,C_2AH_8 , $C_2AH_{7,5}$, C_2ASH_8 and C_3AH_6 also C_4AH_{13} and C_4ACcH_{11} as well as **Acetate** containing calciumaluminum 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 *Calciumonochloroacetate*.

Keywords: *Acetates, Additives, HAC, Heatflow Calorimetry, Hydration*

STEAMBOAT AGGREGATES: A CASE HISTORY USING ADVANCED MICROSCOPY TO IDENTIFY POTENTIALLY REACTIVE MINERALS IN HIGH PERFORMANCE CONCRETE AGGREGATES – RENO, NEVADA

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ABSTRACT

Young basalts from quarries near the Steamboat Springs area have been used as high-performance concrete aggregate in the I-580 Connector Project. Due to the prominence and expense of this project, the NDOT had increased the design life of the project to 100 years and had reduced the allowable mortar bar expansion values to 0.1 percent at 14 days.

The aggregate underwent petrographic examination and was subjected to testing for potential alkali-silica reactivity (ASR) in accordance with ASTM C1260, the accelerated mortar bar method. Initial petrographic results did not indicate the presence of anomalous silica; however, the results of the first series of accelerated mortar bar testing resulted in a 14-day expansion value of 0.34, exceeding the project specification by a factor of 3. Additional mortar bar testing with varying combinations of cement and cementitious materials was performed.

In an effort to determine the cause of the ASR, advanced microscopy techniques were applied. This analysis determined the presence of microcrystalline silica coatings in the aggregate. Based on these results and on accelerated mortar bar expansion test results, it was recommended that a 35 percent Class F Flyash replacement should be used as a mitigation method for potential ASR for Steamboat Aggregates. This level of flyash replacement provided a level of ASR expansion (less than 0.1 percent) that was acceptable to the Nevada Department of Transportation.

REACTIVITY OF CALCIUM CARBONATE FILLER IN TERNARY BINDER SYSTEMS: A CASE HISTORY

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ABSTRACT

The physical properties of a preblended powdered grout for the execution of a new technique for broadband installation (onedaydig) have been strongly affected by the different nature of the filler.

This grout is based on a ternary binder system composed by CAC, OPC and a low content of calcium sulphate

Replacing the quartz filler, contained in the original formula of the grout, with calcium carbonate, a high increasing of early age mechanical strength has been observed.

To better understand the reasons of this behavior, some experiments have been carried on.

Different formulations have been deeply investigated on a morphological point of view by SEM and XRD in order to follow the hydration kinetics.

The Physico-Mechanical and Microstructure Properties of Cement Paste using different cement Types.

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ABSTRACT

In this study the hydration process of sulphate resisting cement (S.R.C.) pastes was compared to that of corresponding granulated slag (G.S.C.) and ordinary Portland cement (O.P.C.). Water of consistency, initial and final setting time, chemically-combined water, free lime contents and compressive strength of the hardened cement pastes were determined. The extent of hydration was followed by the determination of compressive strength of the different cement types cured in 4% $MgSO_4$ solution up to 12 months. Scanning electron microscopy (SEM) technique was carried during the hydration for the different types of cement pastes.

LIME SATURATION FACTOR : NEW INSIGHT

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ABSTRACT

Lime saturation factor (LSF), associated to silica ratio (SR) and alumina modulus (AM) is well known as an useful tool to calculate the raw meal of Portland cement clinker.

Unfortunately, LSF gives little information on the quality of the clinker (% of C_3S or alite). In this paper we demonstrate that LSF is not a linear function of the percentage of C_3S and that the scattering of the relation LSF versus % C_3S is not due to a lack of accuracy in the measurement of C_3S, but can be explained by a variation of the silica ratio and alumina modulus.

Key words: raw mix, silica ratio, alumina modulus, Lime saturation factor, Bogue's formula

RESUME

Le facteur de saturation en chaux (LSF), associé au rapport silicique et au module aluminique sont les principaux paramètres de contrôle permettant de calculer le cru du clinker de ciment Portland.

Malheureusement le LSF ne donne que peu d'informations sur la qualité du clinker (% de C_3S ou d'alite). Dans cette étude, nous montrons que le LSF n'est pas une fonction linéaire du % C_3S et que la dispersion des points dans la relation LSF en fonction de C_3S n'est pas due à un manque de précision des mesures mais peut être expliquée par les variations du rapport silicique et du module aluminique.

DURABILITY OF SULPHATE RESISTANT CEMENT HAVING DIFFERENT BELITE/ALITE RATIOS IN SEA WATER

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ABSTRACT

The kinetic of hydration of sulphate resistant cement having different belite/alite ratios cured in tape water up to 90 days was determined. The durability of cement pastes exposed to sea water up to 9 months was investigated by measuring the bulk density, porosity and compressive strength and the physico-chemical properties from total sulphate and chloride contents. The hydration products and deleterious effect of sea water on the pastes have been determined by using TGA, IR, and SEM equipment. It was found that as the belite content increases in the SRC the durability of cement pastes in seawater is improved.

MICROSTRUCTURAL STUDY TO EVALUATE TREATMENT REPERCUSSION ON AGGREGATE/CEMENT MATRIX INTERFACE IN LIGHTWEIGHT CONCRETE

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ABSTRACT

Cementious composites using vegetables byproducts as aggregates become more required. In this kind of composites, the naturally high hydrophilic capacity of vegetables has direct consequences on concrete performance especially the decrease in mechanical strength and the increase in dimensional variations. Therefore, some treatment processes have been designed to minimize the water retention by vegetables.

Studied lightweight concretes have been prepared using flax shaves like aggregates and Portland cement like matrix.

Before their incorporation, flax shaves have been submitted to coating treatments using many coating substances (mineral substances, biopolymers…). this study deals with the microstructure changes of concrete due to these treatments. The relation between these changes and concrete performance is studied using scanning electronic microscopy (SEM). The influence of these treatments on cement hydration products and aggregate/cement matrix interface is presented.

RESUME

Les composites cimentaires utilisant les coproduits végétaux comme granulats sont de plus en plus demandés. Dans ce genre de matériaux, la forte capacité hydrophile des granulats lignocellulosiques a des conséquences directes sur la performance du produit fini, spécialement sur la diminution de la résistance mécanique et l'augmentation des variations dimensionnelles. Donc, des procédés de traitement ont été mis au point afin de diminuer la capacité de rétention d'eau des granulats lignocellulosiques.

Les matériaux lignocellulosiques sont préparés en utilisant les anas du lin comme granulats et le ciment Portland comme liant.

Avant de les incorporer dans la matrice cimentaire, les anas ont subi des traitements d'enrobage utilisant plusieurs substances d'enrobage (minérales et polymériques…).

Cette étude révèle les changements de microstructure du béton dus aux traitements des anas et la relation entre ces changements et la performance des composites cimentaires en utilisant la microscopie électronique à balayage (MEB). L'influence de ces traitements sur les produits d'hydratation du ciment est présentée ainsi que les changements au niveau de l'interface granulat/matrice avant et après traitements.

EFFECT OF THERMALLY TREATED TEMPERATURE ON CLAY BRICKS (HOMRA) AND FLY ASH COMPOSITE CEMENT PASTES

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ABSTRACT

The influence of high temperature on the phase composition and physico – mechanical properties of clay bricks –fly ash composite cement pastes was studied. The addition of pozzolana such as clay bricks (homra) and fly ash to Portland cement improves the fire resistance due to their reaction with lime liberated from the hydration of ordinary Portland cement paste forming hydration products which precipitate in some of the open pores . The homra pozzolanic cement composed from 80 wt % OPC and 20 wt% homra was substituted by fly ash at 5 , 10, 15 and 20 wt % to prepare composite cement . The cement pastes cured for 28 days in tap water were fired at temperature 105, 250, 450, 600 and 800 ^{o} C with rate of firing 2 ^{O}C /min and soaking time 2 hrs at each temperature. The effect of fire on the cement pastes was investigated from the weight loss , bulk density , total porosity and compressive strength. The phase composition and microstructure were determined using XRD and SEM techniques. It can be concluded that the substitution of homra with fly ash improves the fire resistance of composite cement pastes, i.e. as the fly ash content increases the fire resistance improves. 20 wt % fly ash gives the optimum fire resistance of the pozzolanic cement pastes.

AUTOMATED QUANTIFICATION OF AIR VOIDS IN RCC: IMAGE PROCESSING AND ANALYSIS

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Abstract
This paper reports the characterization of air void systems using image analysis of backscattered electron (BSE) images of roller compacted concrete (RCC) mixes for pavements. Two sets of of comparable mixes were formulated to understand differences in the air void characteristics in non-air entrained and air entrained mixes. Binary segmentation of the sections is used to separate air voids from rest of the phase. Various operations are subsequently utilized to prepare the image for quantification of air void surface area.

It is observed that in RCC mixes, large compaction voids are entrapped during compaction and entraining air requires high dosage of air entraining agent (AEA). Air entrainment enhances the air void size gradation and also increases the total air content in the corresponding mixes (mixes with identical cement contents). However, entraining air comparable to that of conventionally compacted concrete is difficult. Shape factors of different air voids indicate that mixes not incorporating AEA contain much higher fraction of oblate air voids, while an increase in the percentage of improved shape (circular, implying spherical in 3D) occurs with the entraining of air.

The Physico-Mechanical and Microstructure Properties of Cement Pastes containing Silica Fume and Admixture.

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ABSTRACT

The effect of 20% silica fume (S.F) replacements on the blended cement paste properties was investigated. The hydration process of these blended cement pastes were compared to that of corresponding sulfate resisting cement (S.R.C.). Water of consistency, initial and final setting time, bulk density, total porosity and compressive strength of the hardened cement pastes were determined. The extent of hydration was followed by the determination of chemically-combined water, free lime contents and scanning electron microscopy (SEM) technique during the hydration of the hardened cement pastes.

FIRE RESISTANCE OF SILICA FUME-FLY ASH COMPOSITE CEMENT PASTES

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ABSTRACT

This study reports the effect of temperature on the silica fume-fly ash composite cement pastes up to 800 ^{o}C. Pozzolanic cement was prepared from 80 Portland cement with 20% wt silica fume. The silica fume was partially substituted by 5, 10, 15, and 20 wt% fly ash to prepare composite cement. The results revealed that the water of consistency decreases with the fly ash cement due to the high surface area of silica fume. On the other side the initial and final setting time elongated with the fly ash content. This is also due to the low pozzolanic activity of fly ash at early ages. The effect of heat is carried out by determining the ignition loss, bulk density, total porosity, compressive strength for the cement pastes up to 800 ^{o}C. The bulk density of the composite cement pastes increases with the fly ash content. Generally the bulk density decreases at 250 ^{o}C due to the evaporation of some of free and bound water but increases linearly for all cement pastes up to 800 ^{o}C. This is mainly due to the reaction of the silica fume as well as fly ash with the decomposed $Ca(OH)_2$ forming solid-state material such as Ca-silicates or Ca-aluminosilicates. The products of thermally treated composite cement pastes were investigated by using XRD and SEM techniques. It can be concluded that the composite cement with 10-15 wt % silica fume, 5-10 wt% fly ash can be used as fire resistance cement.

Key words: The composite cement, temperature resistance, silica fume, fly ash.

The Uses of Potteries Crushed Waste in Decorative Pottery Tile Manufacture

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ABSTRACT

Crushed pottery generated from potteries sites is usually delivered to landfills for disposal. With the limited landfill space there is an immediate need to explore the possible use of potteries waste as a new material. Today's architectural, design, and building industries is widely using the decorative pottery tiles in commercial, civic, hospitality and residential projects. The main objective of this study is to reuse the potteries crushed wastes to produce decorative pottery tiles, as will as the effect of both organic and inorganic admixtures on its durability and mechanical properties. The mineralogical composition using both x-ray diffraction and scanning electron microscope techniques are also investigated. As a result, it was concluded that potteries crushed waste can be utilized in decorative pottery tile manufacture by taking advantage of low cost and environmental protection.

Keywords: Industrial wastes, potteries, decorative pottery tiles, mineralogical composition, cement, lime, gypsum.

DEF AND ASR IN CONCRETE – A SYSTEMATIC APPROACH FROM PETROGRAPHY

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ABSTRACT

A systematic petrographic examination, involving optical and scanning electron microscopy, x-ray microanalysis, and x-ray diffraction is needed for diagnosing evidence of delayed ettringite formation (DEF) and alkali-silica reaction (ASR) in a concrete. The present article provides an interesting case study of combined occurrence of both mechanisms in a concrete sample, not from the field, but from a left-over concrete pile in a precast concrete plant, which showed spectacular development of cracks from internal expansion due to DEF and ASR. The study diagnosed characteristic microstructural evidence of DEF and ASR, e.g., (a) characteristic patterns of microcracks formed due to DEF and ASR, (b) gaps or peripheral separations around aggregate particles, indicating expansion of paste relative to aggregates, (c) secondary ettringite deposits in gaps and cracks, and (d) alkali-silica reaction gel in cracks, paste, reactive aggregate margins, and voids. The combined microstructural evidence, from both mechanisms, was used to diagnose the occurrence of ASR prior to DEF.

Cementitious Materials Crystal Structure Database for X-Ray Powder Diffraction Analyses[1]

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Abstract

The growing interest in X-ray powder diffraction (XRD) measurements of clinker and cements has resulted in the acceptance of the first standard test method for powder diffraction analysis of clinker and cement ASTM C1365, Standard Test Method for Determination of the Proportion of Phases in Portland Cement and Portland-Cement Clinker Using X-Ray Powder Diffraction Analysis. The resurgence in XRD analysis of cements is partially due to the development of the Rietveld refinement method for multi-phase systems. This Rietveld approach requires qualitative identification of the phases in a sample, crystal structure models that serve as virtual reference standards for those phases along with a comprehensive database of this information before the refinements can proceed. This paper describes the comprehensive database of crystal structures on phases found in clinker and cements and was developed to facilitate the use of XRD analysis of cements. This database is indispensable for qualitative and quantitative analyses of phase compositions using Rietveld refinement of X-ray powder diffraction patterns.

Keywords
Cement, clinker, crystal structures, database, quantitative analysis, Rietveld refinement, X-ray powder diffraction