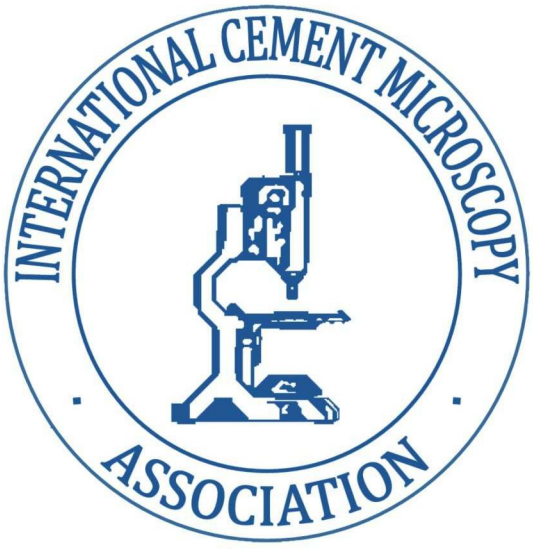
21st INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY



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MICROSTRUCTURAL AND PHYSICO-CHEMICAL CHARACTERISTICS OF SOME AUTOCLAVED BLENDED CEMENT PASTES

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ABSTRACT

Autoclaved specimens made of Portland cement-fine sand blended cement , known in Egypt as El-Karnak cement , containing silica fume and / or by-pass dust (cement kiln dust) were cured hydrothermally at a pressure of 10 atm. of saturated steam for various time intervals of 0.5 , 2 , 6 , 12 and 24 hours . The hydrothermally hardened specimens were tested for their compressive strength , kinetics of hydration , phase composition and microstructure . The effect of addition of silica fume and / or by-pass dust on the improvements of the physico-mechanical and microstructural characteristics of the autoclaved specimens could be explained on the basis of the results obtained in this investigation .

RESISTANCE OF DIFFERENT CEMENTS TO AMMONIUM SULFATE

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ABSTRACT

Five concretes containing a cementitious material content of 350 kg/m^3 were subjected to wetting-drying cycles in a 200 g/L ammonium sulfate solution. The following cementitious materials were used:

- ordinary portland cement,
- blast-furnace slag cement,
- three mixtures of blast-furnace slag cement and calcined paper sludge.

Calcined paper sludge is a reactive pozzolanic material obtained by calcining paper sludge at 650°C.

After 4 cycles of corrosion, only concretes containing blast-furnace slag cement associated to calcined sludge still presented some strength (30 to 37 % of the initial value). These specimens also showed the smallest mass loss after 6 cycles of corrosion.

The microstructure of these concretes was investigated by SEM. Gypsum was present at aggregate-paste interfaces and the size of the crystals depended upon the binder used. C-S-H decalcification was sometimes observed.

INFLUENCE OF POLYMER TYPE ON THE STRUCTURE OF POLYMER MODIFIED CEMENT MORTAR

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ABSTRACT
The addition of polymer emulsion and biocides to the fresh mixture to improve the durability of concrete sewer pipes against biogenic sulphuric acid corrosion is investigated.

The paper deals with the results of the preliminary tests where the behaviour of polymer modified cement mortar is investigated using different types of polymer emulsion. The mechanical properties (compressive strength, bending strength) as well as the physical properties (density, porosity) are determined on mortar beams. Two different curing conditions are used: standard curing, in which a wet curing period is followed by a dry curing period, and dry curing conditions, which is in favour of the polymer film forming.

By means of SEM the influence of the polymer emulsion on the structure of the mortar is investigated. Special attention is drawn to the formation of a film and to the positioning of the polymer film or polymer particles in relation to the aggregates and pores. The SEM-study is carried out on broken samples as well as on acid-etched samples. The relation with the MFFT (minimum film forming temperature) of the polymer emulsions is investigated.

Thermal activation and mechanical properties of kaolinite /quartz mixtures

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Abstract : Kaolinite is used to obtain artificial pozzolans from its calcination products. Unfortunately, it does not occur in natural conditions as a pure phase but always mixed in various proportions with many secondary minerals. Among them, quartz is one of the most common associated minerals.

Natural clays, mainly composed of kaolinite and quartz, have been burnt at 600, 700 and 800°C and the calcination products have been characterized by XRD supplemented by SEM observations and granulometric analysis, in order to determine the quartz influence on kaolinite calcination. Mechanical strength tests on portlandite/calcination products pastes have been performed and compared to those obtained on pastes prepared from quartz added to burnt kaolinite. Results allow to ascertain the role of quartz as a function of the burning temperature, the burning time, the water/solid ratio on the development of mechanical strength.

WEATHERED CLINKER, CLEAR BELITE, ALKALI-AGGREGATE REACTION TESTING, AND SIMULTANEOUS REFLECTED- AND TRANSMITTED-LIGHT OBSERVATIONS

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ABSTRACT

This brief paper attempts to briefly describe the results of four separate investigations. The items reported are: (1) The alteration of portland cement clinker by weathering is described. Pseudomorphic hydration of the calcium silicates, forming “inner product,” was locally observed to take place before hydration of C_3A. Calcite crystallization, fully developed in the altered clinker, is shown to be largely destructive. (2) Slow cooling in a cement kiln is inferred from the observation of clear belite with lamellar extensions, using ICMA Sample Exchange Clinker #59 in polished thin sections and powder mounts of clinker treated with a potassium hydroxide-sugar solution. (3) A 28-day petrographic method for identification of reactive aggregates in a modified ASTM C 1260 mortar, utilizing No. 30-mesh (0.60 mm) materials is presented. (4) A technique of microscopical observation utilizing polished thin sections or “half sections” with simultaneous reflected and transmitted light on the same field of view of fly ash and clinker raw feed, thus providing optical data from both modes of observation, is offered.

COMPARATIVE STUDY AMONG THE POSSIBLE BINDERS FOR THE RESTORATION OF HISTORICAL BUILDINGs AND IN PARTICULAR OF THE SAN FRANCESCO BASILICA IN ASSISI

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ABSTRACT:

We have compared different binding systems not only from the analytical point of view but also from the physical-mechanical properties in order to find the connections between the characteristic of products and their performances, even and above all, as a guarantee of durability of the products for the restoration of ancient buildings.
Our analyses were particularly focused on the calcium hydroxide present in different forms in all the binders.
Thanks to our studies onto the original materials used for the building of the S. Francesco Basilica, we verified that the action of the calcium hydroxide contained into the mortars sometimes can be "dangerous" for the structure of the bricks.
The binding systems that we proposed for the restoration, even if they contain the calcium hydroxide, behave in such a way to reduce its concentration to zero, practically one week after the application.
According to these considerations the proposed systems are preferable to the normal cementitious or hydraulic lime based binders. In fact, they show the best characteristics of the first ones (good mechanical resistance, short setting time) without their negative properties (high elastic modulus, etc); while, compared with the second ones, they have an adjustable setting time, a higher mechanical strength and above all the calcium hydroxide disappears after few days.

CEMENT CLINKER STUDIED BY PROTON AND ELECTRON INDUCED X-RAY EMISSION

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ABSTRACT

Planar samples of sulfate resisting Portland cement clinker have been investigated by energy dispersive analysis of emitted X-rays in two types of experiment, using (i) a standard scanning electron microscope, SEM (0.02 MV electron beam with < 0.1 μm diameter, ≈ 3 μm penetration depth, ≈ 1000 ppmw element detection limit), and (ii) a nuclear microprobe, NMP (2.55 MV proton beam with ≈ 10 μm diameter, ≈ 70-80 μm penetration depth, ≈ 1 ppmw element detection limit). Spectra were collected from clinker areas, approximately 500 x 350 μm (SEM) and 380 x 380 μm (NMP) in size, and apart from the main elements Ca, Si, Al, Fe, K, S, significant peaks were observed for the minor elements Mg, Ti, Cr, Mn, Ni (SEM) and Ti, V, Cr, Mn, Ni, Cu, Zn, As, Sr, Y, Zr, Ba (NMP). The increase in the number of observed minor elements reflects the lower detection limit of the NMP compared to the SEM. By elemental mapping of the major elements; alite, belite, oxide and sulfate interstitial material, and pores can be readily identified. The average size of the alite and belite crystals was around 50 μm in the clinker. The spatial resolution in the SEM maps was higher than in the NMP maps, due to the smaller probe diameter and penetration depth of the electron beam. Mapping of minor clinker elements using the NMP appears to be limited by the spatial resolution relative to the size of the clinker crystals. Strontium was shown to be enriched in the belite phase by NMP mapping.

DILATATION AND PHASE DEVELOPMENT IN PASTES OF ALUMINATE CEMENT, PORTLAND CEMENT AND β-CALCIUM SULFATE HEMIHYDRATE

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ABSTRACT

Blends of aluminate cement, ordinary Portland cement and β-calcium sulfate hemihydrate were mixed with water, at a water to solid weight ratio of unity. The dilatation during the hydration process was registered continuously by measuring the change in length of a rectangular paste rod as a function of time. The hydration kinetics was investigated in-situ on reacting pastes by synchrotron X-ray powder diffraction, and by X-ray powder diffraction of samples where the hydration had been stopped with acetone.

During the first 20 minutes there was a 0.5‰ expansion followed by a one hour period without change in the dilatation. Then the paste started to expand during three hours to a total of 5.5‰. During the initial stage of the hydration, gypsum formed in the system and in addition some primary ettringite. The ettringite formation then stopped and the amount of ettringite was constant. The gypsum content reached a maximum and was then replaced by secondary ettringite.

The ettringite formation and expansion show similar behaviour and take place over the same period of time.

INTERFERENCE TECHNIQUES AND THEIR APPLICATION IN CEMENT MICROSCOPY

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ABSTRACT

Polished cross-sectional surfaces of Portland cement and clinker are routinely examined using normal-incidence reflected light optical microscopy. Use of a chemical etch imparts variation in color and texture among and within the various crystal phases which are readily realized using normal-incidence reflected light. Interference techniques have shown utility for examining subtle variations in surface topography in other fields. The intent of the present article is to introduce a particular optical inference technique, Nomarski Differential Interference Contrast Microscopy, and to illustrate through examples the potential usefulness of this technique for examining chemically etched, polished surfaces of cement and clinker materials.

INTERACTION OF HYDRATING CEMENT WITH PETROLEUM INDUSTRY SPACER FLUIDS

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ABSTRACT

This paper presents the results of a study to examine the interaction between hydrating cement and various spacer fluids used in the petroleum industry. Spacers and chemical flushes are used in oil and gas well cementing operations for several reasons. They are used to separate the drilling fluids from the cement to prevent incompatibility, to aid in removal of the drilling fluid, and to leave all surfaces water-wet to enhance bonding with the cement.

Particular emphasis in this investigation was placed on spacers containing blends of surfactants used with synthetic-based drilling fluids. Synthetic-based systems are being widely used in offshore operations to allow the environmentally accepted discharge of drill cuttings on-site. The surfactants allow the water-based spacers to be compatible with the synthetic-based fluids and invert them to a water-wet condition so cement can bond to any residue that might remain.

This investigation examines (1) the microscopical interaction between the fluids and (2) the compressive strength compatibility of the fluids. The microscopical examinations reveal that some of the spacers appear to increase the formation of ettringite. The two clay-based spacers containing the blend of several surfactants both exhibited large "nests" of ettringite crystals. Three other spacers containing singular surfactants exhibit a notable amount of ettringite, but considerably less than the two with heavy surfactant loading. The pozzolan-based spacer contains the least amount of ettringite and is the only one that does not contain surfactants. Compressive strength determinations show the pozzolan-based spacer (no surfactant) to have the highest compressive strength of the contaminated slurries. The spacers with the heavy surfactant loading are not the ones with the lowest compressive strength however. After curing at 160°F for 120 hours there is virtually no difference between them and uncontaminated cement. The spacer marked clay 2 contains a small amount of retarder and the emulsion-based spacer contains a moderate amount of retarder. This explains the lower compressive strengths for those specimens. The lower compressive strengths of the samples contaminated with the solvent-based spacer may be due to the presence of a mutual solvent in the system which allows the solvent to essentially become part of the cement matrix. More research is needed to examine the possibility of surfactants causing enhanced ettringite formation during cement hydration.

QUANTITATIVE RIETVELD PHASE ANALYSIS OF HYDRATED PORTLAND CEMENTS: I. QUANTITATIVE ANALYSIS OF SYNTHETIC AFm AND AFt PHASES

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ABSTRACT

Ettringite ($C_3A \cdot 3CaSO_4 \cdot 32H_2O$), Monocarbonate ($C_3A \cdot CaCO_3 \cdot 11H_2O$), Monosulfate ($C_3A \cdot CaSO_4 \cdot 12H_2O$) and Portlandite $Ca(OH)_2$ are the main crystalline phases of hydrated Portland cements. X-ray diffraction pattern analysed by the Rietveld method enables quantitative determination of crystalline and amorphous phases contained in hydrated Portland cements.

Ettringite, Monocarbonate and Monosulfate were synthesised under CO_2-free conditions. The X-ray pattern of this phases showed preferred orientation. To minimise the preferred orientation the samples were prepared with the side-loading technique. In the case of Monosulfate a paste sample has to be prepared to avoid preferred orientation. Standard data of Ettringite, Monocarbonate and Monosulfate were refined on basis of the X-ray pattern nearly free from preferred orientation.

Physical mixtures of defined amounts of this three phases were made to demonstrate the precision and reproducibility of the quantitative Rietveld analysis. Although the mixtures showed preferred orientation the Rietveld software was able to correct this effect by using the March Dollase function. Therefore it was possible to quantify such model mixtures with a precision of ±3 wt.-%.

100 YEARS' OLD RENDERING MORTARS CHARACTERIZATION AND MICROSTRUCTURE

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ABSTRACT

Rendering mortars of the *Cruz e Souza* Palace in Florianópolis - SC - Brazil, built in the beginning of the century, are subjected to serious damage like detaching, crumbling and cracking.
The purpose of this work is to identify the rendering mortars components to reproduce them, if possible, in the damaged areas. Samples from two healthy regions of the rendering were studied: the first one from an ornament, a type of console which sustains the roof eaves of the Palace and the other one from a masonry rendering. Samples were characterized by X-Rays Diffraction (XRD), Differential Thermal Analysis (DTA) and Scanning Electron Microscopy (SEM) with Energy Dispersive Analysis X-Rays (EDAX). XRD and DTA showed, in both cases, that the principal binder is a calcium carbonate coexisting as calcite and vaterite for the first sample, and only as calcite for the other one. SEM showed the two morphologies of calcium carbonate and the existence of an amorphous calcium hydrosilicate for the first case. An interpretation about the origin of these renderings is then presented.

MICROSTRUCTURE OF NEW FLY ASH-BELITE CEMENT MORTAR: CHANGES PROVOKED BY SULPHATE, CHLORIDE AND SODIUM IONS

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ABSTRACT

Characterization of the microstructure of mortars fabricated with new fly ash-belite cements and changes provoked by a potential aggressive solution (NaCl (0.5M)+Na_2SO_4 (0.5M)) are discussed. The results showed a preferred combination of sulphate versus chloride with the solid phase to form non-expansive sulphated compounds of ettringite (AF_t) type: $Ca_6Al_2(SO_4,SiO_4,CO_3)_3(OH)_{12}.26H_2O$ and monosulpho aluminate (AF_m) type: $4CaO.0.9Al_2O_3.1.1SO_3.0.5Na_2O.16H_2O$. Consequently, the microstructure becomes more compact, decreasing the porosity and increasing the mechanical strength. The experimental methodology was carried out according to the Köch-Steinegger test by immersing mortar specimens in the aggressive solution during 180 days at the temperature of 21ºC±2ºC. Similar specimens were immersed in demineralized water as reference.

AN ANALYSIS OF THE VARYING COMPOSITION OF CLINKER MINERALS WITHIN AND BETWEEN CEMENT CLINKERS, WITH SOME OBSERVATIONS ON OPERATIONAL IMPLICATIONS.

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ABSTRACT

Seven clinker samples have been examined using scanning electron microscopy and energy dispersive x-ray microanalysis. The main phases in each clinker have been characterised in terms of their oxide analysis. The quantities of minor oxides within each phase have been measured and their relationships to the structure of the crystals examined. An appreciable proportion of the Fe_2O_3 and Al_2O_3 within alite crystals has been demonstrated to be present as inclusions of liquid phase and not as replacements of the main oxides. The relationship of alkali and sulphate to belite crystals has been demonstrated to vary depending on the chemistry and firing conditions within the cement kiln.

EFFECT OF SOME ADMIXTURES ON THE HYDRATION OF SILICA FUME AND HYDRATED LIME

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ABSTRACT

The effects of sodium salt of naphthalene formaldehyde sulfonic acid and stearic acid on the hydration of silica fume and $Ca(OH)_2$ have been investigated. The hydration was carried out at 60 C and by using W/S ratio of 4 for various time intervals namely, 1, 3, 7 and 28 days. Two ratios of the admixtures were used (2 and 5% by weight of solids). The results of the hydration kinetics show that both admixtures accelerate the hydration reaction of silica fume and calcium hydroxide during the first 7 days. Whileas, at 28 days of hydration there is no significant effect. Generally , most of free calcium hydroxide seems to be consumed after 28 days. In addition, the phase composition as well as the microstructure of the formed hydrates were examined by using X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) respectively.

EFFECT OF SUPERPLASTICIZER ON ETTRINGITE FORMATION

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ABSTRACT

The suspension hydration of C_3A with gypsum (in the molar ratio of 1: 3) was investigated at room temperature and W/S ratio of 4. The hydration was carried out in presence of 0, 1 and 3% of sodium salt of naphthalene formaldehyde sulphonic acid condensate and the mixes were designated as I, II and III respectively. The only hydration product formed in presence and absence of the superplasticizer was ettringite.

The rate of ettringite formation was retarded by the presence of the superplasticizer. This effect was more pronounced, at high dosage of the superplasticizer, only during the first 24 hours. The presence of sulphonated naphthalene formaldehyde caused a decrease in the degree of crystallinity and size of the formed ettringite crystals, and as the percent of the admixture increased, the crystal size was decreased. In addition, there was an interaction between the used superplasticizer and the formed ettringite as indicated from IR analysis.

MICROSTRUCTURAL INVESTIGATION ON THE ROLE OF MODIFIERS IN THE STABILIZATION OF DIFFERENT POLYMORPHS OF SILICATE PHASES OF OPC CLINKER

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ABSTRACT

OPC clinker samples were prepared with selected modifiers that have been reported to have significant effect on the formation of clinker phases. The role of these additives in the development and modification of the clinker phases has been investigated using optical microscope, SEM-EDS, XRD techniques. Chemical separation techniques have been employed to understand the partitioning of the elements such as boron, manganese and chromium in presence of potassium in the silicate and non silicate fraction of the clinkers. The paper also evaluates the effect of these element on the microstructure in terms of phases assemblages, crystal size and morphology and polymorphism of silicate phases. The study indicates the effectiveness of the elements on alite formation can be graded in the order Mn_2O_3 > Cr_2O_3 > K_2O >> B_2O_3 whereas boron addition substantially stabilizes the belite formation.

HYDRATION OF CLASS G OILWELL CEMENT AT 20°C AND 5°C WITH CALCIUM FORMATE

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ABSTRACT

Oilwell cements are used in the oil industry in exploration and in the production of oil and gas. Their primary use is to seal the annular space between the walls of a borehole and secure the steel casing that lines the well. During the casing cementing of well sections at or near the surface under cold conditions, where the temperatures may be as low as around 0-5°C, typically in permafrost regions such as found in the Arctic, the cement hydration reactions are slowed down and chemicals like 'accelerating' admixtures, are used to promote hydration. These admixtures adequately accelerate the cement hydration reactions so that the cement performs its intended function, whether this is chemical, physical or both. The documentation of hydration aspects of oilwell cements in the literature is scarce since these types of cement do not fall into the mainstream of cement research. Frequently, extrapolation of data from construction Portland cements is done and applied to oilwell cements. This is not wise since oilwell cements and construction cements are chemically and physically different and can lead to the potential of a bad cementing job. Calcium formate ($Ca(HCO_2)_2$) had not previously been studied at a fundamental level with oilwell cements. This omission has now been remedied.

The hydration aspects of Class G oilwell cement at 20°C and 5°C with additions of $Ca(HCO_2)_2$ have been investigated by applying several techniques: isothermal conduction calorimetry, scanning electron microscopy with backscattered electron imaging, X-ray powder diffractometry, thermal analysis, infrared spectroscopy and the development of compressive strength. The cement was mixed with distilled water, with a water-to-cement ratio of 0.44.

The hydration of the oilwell cement in the presence of $Ca(HCO_2)_2$ at 20°C is different from that at 5°C. $Ca(HCO_2)_2$ influences the development and morphology of the C-S-H. The times at which AFt and AFm phases - ettringite (and formate analogues) and monosulphate (and formate analogues) respectively - are strongly dependent upon the dosage and temperature of curing. The acceleratory effect of $Ca(HCO_2)_2$ is spread over a long period of time and it is continuously contributing to the overall hydration of the cement. It appears that the $Ca(HCO_2)_2$ enhances the hydration of the ferrite phase. There is some evidence that the formate ion is itself becoming incorporated in the hydration products like C-S-H and contributing to the overall properties of the cement paste. In some instances the addition of $Ca(HCO_2)_2$ shows a retardation effect, but the compressive strength of the paste is increased relative to the control. Here the $Ca(HCO_2)_2$ acts as a 'strengthener' rather than an accelerator. Its behaviour is different from that of calcium chloride and sodium aluminate. There is no universal mechanism of acceleration of cement hydration. The actual mechanism depends upon the particular accelerating admixture, dosage, the time of hydration and the curing temperature.

MICROSTRUCTURE OF CALCIUM SILICATE HYDRATES DOPED WITH SOME ADMIXTURES

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ABSTRACT

The microstructure of hydrated calcium silicates formed at ambient temperatures as well as under the saturated water vapour at 200°C was observed by SEM. The lime-quartz mixtures with CaO to SiO_2 molar ratio of 1.00 and 0.83 were prepared. The effect of some admixtures, such as $CaCl_2$, $AlCl_3$, $CrCl_3$, Na_2CrO_4, NaOH, $Al(OH)_3$, $Zn(NO_3)_2$, $Pb(NO_3)_2$ and $CdCl_2$ was investigated. At CaO/SiO_2 =0.83 or 1.00 in the samples cured in hydrothermal conditions the transformation of C-S-H to tobermorite was generally observed. The crystallization of xonotlite was generally inhibited by the presence of admixtures and perhaps by use of quartz. At higher content of some heavy metals containing admixtures the formation of well developed C-S-H or tobermorite crystals was hindered in some cases but at lower content of admixtures or with no admixtures the better shaped forms were produced. At the prolonged hydrothermal curing (200°C/48h) the fibrous and acicular crystals were replaced by smooth, large plates, polyhedral or almost circular, forming spherical intergrowths or three-dimensional network. In many samples the simultaneous occurring of different morphologies as well as the transitions between the particular forms were observed.

SEM-EDS: SEMI-QUANTITATIVE APPLICATION TO INVESTIGATE THE CAUSE OF «COLOR AND STAINING DEFECTS» OF CEMENT-BASED MATERIALS - CASES STUDY.

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ABSTRACT

Scanning Electron Microscopy fitted with Energy Dispersive Spectrometer is a very useful tool, especially in the field of concrete failure.
Among various qualitative applications, the most common ones concerns the chemical pathologies of concrete: investigations about alkali-aggregate reaction and secondary ettringite formation are among the most numerous.

Nevertheless the technique is ideal, valuable and probably one of a kind when used in semi-quantitative way to make original, useful and reliable investigations with regards to particular color or staining disorders likely to occur during cement-based materials manufacturing.

The paper reports how the results of a semi-quantitative SEM-EDS analysis could contribute to provide some explanation about the possible cause of unexpected coloring defect of concrete masonry blocks and surface staining of cement tiles.

DELAYED ETTRINGITE PRECIPITATION VIA ACID ATTACK

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ABSTRACT

Primary ettringite formation is inhibited by elevated temperatures (above 60º-70ºC) or high alkali content medium. In the first case, if concrete is subsequently kept in water or humid conditions at ambient temperature, ettringite may crystallize in the paste. This type of ettringite is called "delayed ettringite" and traditionally, is a tern used to denote the formation of ettringite in concrete that has been cured at high temperature. In the present paper it is discussed that, when high alkali content is the cause that impedes primary ettringite formation, acid attack on concrete leads to ettringite crystallization in the paste.

PITFALLS IN SCANNING ELECTRON MICROSCOPY (SEM) STUDIES

by

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ABSTRACT

Use of a scanning electron microscope (SEM) and energy dispersive X-ray elemental analysis (EDS) to identify components in materials such as cement paste or concrete is a fairly new type of identification procedure. The identification is based on the presence of the main peaks of particular elements on the spectrum and the ratios among these peaks. However, the size of the analyzed area plays an important role, failure to understand the cementitious systems and the instruments capabilities can lead to misleading and erroneous conclusions. For correct interpretation of the obtained data a good geological background and a knowledge of cement chemistry is needed. In the case of failure investigation, SEM studies should be considered as an adjunct analysis following petrographic observations and engineering evaluations of the particular structure.

EFFECT OF SILICA FUME ON MASONRY MORTAR MICROSTUCTURE AND PROPERTIES

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ABSTRACT

The functions of mortars which are used for brick and block laying in masonry walls are: (i) to bond units of masonry; (ii) to distribute loads; (iii) to absorb deformations; (iv) to stamp joints.
Several additions, mineral and organic, are used in mortars, such as pozzolanic materials, cementicious materials and polymers. The literature about the use of additions in masonry mortars (cement/lime/sand mixes) is scarce; usually, studies are about concrete mortars.
The purpose of this work is to study the effects of the substitution of 10 and 20 % of the binder cement-lime by silica fume in a 1:1:6 (cement/lime/sand mix proportion by volume) masonry mortar on microstructure (SEM and EDAX) and on engineering useful properties (consistency, shrinkage, compressive and tensile strength and deformation modulus).
SEM observation shows that with silica fume, a densification of the interface paste-aggregate occurs as it is observed in concrete mortars, C-S-H type III is formed at early ages and C-S-H types I and II are formed after seven days.

OLDHAMITE: NOT JUST IN METEORITES

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ABSTRACT: Oldhamite, (CaS) has been recognized as a constituent of meteorites for over one hundred years. Aside from meteorites, and the occasional volcanic glass, blast furnace slag (BFS) is probably the most common source of oldhamite on planet Earth. The oldhamite in the slag aggregate examined in this study was mostly present as dendritic inclusions within melilite crystals. Thin sections prepared from concrete pavements incorporating BFS aggregate show evidence of oldhamite dissolution. Simplified chemical models suggest that oldhamite in contact with cement pore water may undergo dissolution.

Investigations on phase development in autoclaved calcium silicate building products (autoclaved aerated concrete)

by

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ABSTRACT

The production of autoclaved construction materials is based on the prefabrication of parts which are autoclaved later on. Contrary to cementitious based construction materials the fabrication of lime-based materials is not possible without autoclaving at elevated temperatures for some time. Due to the different raw materials, amounts of materials, applied temperatures and water pressures different amounts of calcium silicate hydrates are formed.
The purpose of this work was to describe the influence of changes in the compositions of the raw materials and the development of the various hydrates which are formed. Also the influence on physical properties of the autoclaved materials is shown.

SEM INVESTIGATION OF MICROBIAL CALCITE PRECIPITATION IN CEMENT

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ABSTRACT

Scanning electron microscopy (SEM) is shown effective in documenting the role of bacteria in microbiologically-induced mineral precipitation. In this work, microbial plugging of artificially-cracked cement mortar beams was studied using *Bacillus pasteurii* combined with different filling materials. After curing for 28 days, specimens were fractured and the interface between the filling material and cement mortar was examined using SEM. Well-formed crystals, measuring 200 μm or more in length were observed as precipitates binding particles of the filling material and binding filling material to the crack wall. Energy-dispersive X-ray spectra of the crystals show only Ca, indicating that the precipitated material is calcite ($CaCO_3$). Furthermore, many calcite crystal faces show hollow, rod-like impressions of *B. pasteurii*, where bacteria in contact with the calcite interfered with normal crystal growth. These microscopic observations serve to confirm the mechanism of microbial calcite precipitation in cement.

Keywords: SEM, cement, calcite, *Bacillus pasteurii*

EFFECT OF COOLING RATE OF CLINKERS ON THE STATE AND REACTIVITY OF C_3A

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ABSTRACT

Following the reburning in a laboratory oven, two commercial clinkers of different chemical and mineral composition were cooled quickly (in water) and slowly (cooling down in a furnace).
The mineral composition of the laboratory-cooled and plant-cooled clinkers, calculated with the Bogue method and with the selective dissolution method, was compared. X-ray diffraction and optical microscopy methods were used to study the crystal modifications and forms of C_3A, and the state of the ferrite-mixed crystal series.
The relationships between the solubility, reactivity and cooling rate of C_3A were investigated. It was shown that the cooling rate has a significant effect on the state, crystal modification (quick cooling results in "proto" C_3A; slow cooling in monoclinic form), and reactivity of C_3A found in the clinker and on the sulfate expansion of cements produced from clinkers.
It has been found that in commercial clinkers C_3A is present as a mixture of crystal forms of various phases and modifications.
The formation of the crystal modification of C_3A is basically determined by the incorporation of alkali-oxides. The incorporation of alkalis at the same time is the function of the cooling rate, therefore both actions have a combined effect on the changes in reactivity and sulfate expansion of C_3A and/or of clinkers.

Study of Microstructure of Concrete by AC Impedance Technique

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Abstract

Mechanical properties and durability are greatly influenced by pore structure of the concrete. A new model of connected capillary network has been proposed to describe the pore structure of the concrete. AC impedance measurement was performed on the specimens and the result of frequency response was plotted on a complex plane (i.e. Nyquist plot). The typical experimental curves are of Randles' type which can be characterized by four parameters: the resistance of pore solution R_{ct}, the interface capacitance C_d, the charge transfer resistance R_{ct} and the coefficient of diffusion resistance σ. The four parameters correspond to the parameters of connected capillary network describing the pore structure of concrete.

The method has been used to measure the regional distribution of microstructure and it is possible that the change of the microstructure in different region of a large specimen can be detected by this method. A specimen of 10cm×10cm×50cm was tested by a probe on different position and direction with the result that a three-dimensional distribution of the microstructure of the concrete can be shown.

The method is non-destructive and may be applied in situ.

Fungal Growth Measurement by ESEM in four different mortars and proposal of a biodeterioration mechanism

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ABSTRACT

Growth of the fungi *Cladosporium sphaerospermum* was observed on four different types of mortar incubated at 25^{o} C. Two mortars were composed of cement, lime and Brazilian standardised sand, the others being of industrial origin. Environmental scanning electron microscopy (ESEM) allowed the observation without any treatment performing a quick and efficient analysis avoiding artefacts, imaging micro-organisms and mortar the in the hydrated state. The mortar pH had a significant effect upon fungal growth. It was possible to propose a biodeterioration mechanism for mortar composed of cement, lime and four fractions of standardised sand 3 months after inoculation with fungi.

CRYSTALLISATION AND MORPHOLOGY OF GYPSUM STUDIED BY SYNCHROTRON X-RAY DIFFRACTION AND SCANNING ELECTRON MICROSCOPY

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ABSTRACT

Experiments were performed to investigate the possibility of recording both the crystallisation kinetics and the resulting crystal shape and texture, on the same calcium sulfate specimen by a combination of synchrotron X-ray powder diffraction and scanning electron microscopy techniques.

Calcium sulfate hemihydrate produced from analytical grade calcium sulfate dihydrate was mixed with water and small amounts of setting modifiers, the paste was drawn into a glass capillary and then mounted on the diffractometer. During the hydration X-ray spectra were collected for three minutes. These data were then converted to intensities as a function of time by integrating the area under selected peaks, one for the hemihydrate and one for the dihydrate. After the samples had been dismounted from the X-ray equipment the glass capillaries were crushed in acetone. Specimens for microscopy were prepared from these samples and later studied in a JSM-840A scanning electron microscope.

The relative X-ray intensities were utilised to follow the dissolution of hemihydrate and formation of dihydrate as a function of time. The time for complete reaction increased in the following order depending on the additives: 5% gypsum < 5% gypsum and 0.25% citric acid < 0.5% gypsum < 0.5% potassium sulfate < no additives < 0.25% citric acid. From the microscope pictures it could be seen that when gypsum was added the material was much more homogeneous, due to the increased number of nucleation sites. It could also be seen that when citric acid was added the amount of thin twin crystals increased. Otherwise the gypsum crystals were normally needle shaped.

MECHANISM OF SETTING OF MAGNESIA-PHOSPHATE CEMENTS

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ABSTRACT

Magnesia-phosphate cements are chemically bonded ceramics formed by the reaction between dead-burned magnesia (MgO) and mono-ammonium phosphate ($NH_4H_2PO_4$). They are used for rapid repair since the 1980s, but the mechanism of setting is not yet well understood.

When in contact with mixing water, the mono-ammonium phosphate (MAP) quickly dissolves to reach saturation while magnesia begins to be wetted. The kinetics of wetting depends upon the surface of magnesia, which is influenced by the temperature of calcination during its preparation. The important pH drop observed during the dissolution of MAP leads to the dissociation of magnesia according to an acid-base reaction which needs the adsorption of three molecules of water.

Once in solution, Mg^{2+} ions react with molecules of water to form $Mg(H_2O)_6^{2+}$ complexes. These compounds can substitute to molecules of water during adsorption on magnesia surface, more specially as their concentration increases. When such complexes are adsorbed, the two supplementary molecules of water needed for dissociation can no longer come close to the surface and, therefore, the complexes gradually recover the surface of magnesia.

Thus, PO_4^{3-} and NH_4^+ ions can approach to the surface and form with $Mg(H_2O)_6^{2+}$ complexes still in solution, a struvite network due to hydrogen bonds.

This mechanism is proposed, based on SEM observations (HITACHI S800 - high resolution).

SPECIMEN PREPARATION for SCANNING ELECTRON MICROSCOPY[1]

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Abstract

Microscopy plays an important role in the examination of cementitious materials. Optical and electron-optical techniques allow examination of microstructural details with sub-micrometer definition. The increased application of scanning electron microscopy in cement and concrete investigations has brought attention to differences in preparation techniques. The success of these investigations is, in part, influenced by the type and quality of specimen preparation. In particular, backscattered electron and X-ray imaging modes are influenced by the specimen surface characteristics, with the ideal surface being highly polished. Saw-cut surfaces that have not been epoxy-impregnated, nor polished, are not representative of the true microstructure, and are difficult to examine and interpret without bias. Sawing creates a series of fractures, which are enhanced with subsequent drying shrinkage. Particulate matter from the sawing is also deposited on the surface. These effects combine to present a surface that is not well suited for any type of microscopy and that is substantially different from the true concrete microstructure. Polished epoxy-impregnated surfaces are relatively simple to prepare and allow the researcher to avoid the above-mentioned difficulties. Claims that this procedure alters, or 'smears', the microstructure have not been substantiated. Procedures developed in our laboratory for preparation of polished sections of clinker, cements, and hardened portland cement concrete preparations are presented here.

PHASE COMPOSITION OF CLINKER MEASURED BY MICROSCOPY COMPARED WITH QUANTITATIVE X-RAY DIFFRACTION (RIETVELD) AND BOGUE RESULTS

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ABSTRACT

Fifteen different clinker have been examined by optical microscopy as well as quantitative X-ray diffraction by Siroquant (QXRD), and the results for the major clinker phases have been compared to the results from Bogue estimations.

The clinker samples originate from a wide range of kiln types and the chemical compositions show large variations in LSF, Ms and Ma as well as minor components.

The QXRD method, using Rietveld, has been used on the clinker and on the liquid phase residue after selective dissolution. The same refinement program has been used for all the clinker, so the method can be used routinely in the laboratory.

The general experience that Bogue often underestimates the content of alite and overestimates the content of aluminate has been confirmed. When the clinker contains mainly orthorhombic aluminate an even higher alite content is noted for 4 of the clinker samples.

The QXRD method shows good results concerning aluminate and ferrite, the two phases, which are difficult and sometimes impossible to measure by microscopy. However more work on the sample preparation or XRD pattern refinement needs to be done in order to achieve good results for alite and belite when such a variety of clinker and crystal sizes are included. However, when production problems need to be solved, optical microscopy still has the advantage.

LONG-TERM INFLUENCE OF ALKALINE EARTH SILICATE ADMIXTURES ON THE MICROSTRUCTURAL DEVELOPMENT OF STRUCTURAL CONCRETE

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ABSTRACT

Specimens from a Pennsylvania Turnpike bridge structure have been examined as part of a study to characterize the physical and chemical properties of structural concrete bridge panels that have been in service for twenty-five years. A remarkable contrast exists between one bridge that was constructed with concrete formulated with the use of an alkaline earth silicate admixture and the identical concrete formulation but without the use of the admixture. While the structure with the admixture appears macroscopically to be only a number of years old, the structure without admixture appears macroscopically deteriorated. On a microscopic scale, a significant difference appears between the two microstructures; one, with the admixture, being dense and continuous and the other, without the admixture, somewhat porous and discontinuous.

INFLUENCES OF SEPARATE AND INTERGRINDING THE RAW MATERIALS OF BLENDED CEMENTS ON HYDRATION

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ABSTRACT

In this study, a portland cement clinker, a natural pozzolan and a granulated blastfurnace slag were used to obtain interground and separately ground blended cements with 25 % mineral additive. All the cements (two natural pozzolan added cements, two blastfurnace slag added cements and a portland cement) had 3500 ± 100 cm^2/g Blaine fineness. Cements having the same composition but produced by intergrinding and separately grinding were compared from compressive strengths and microstructure of hydration points of view. For this purpose, cement pastes were investigated by SEM at 2, 7, 28 and 90 day and compressive strengths of standard mortars with these cements were determined at these ages. Also the shape and size of the cement and mineral additive particles were investigated by SEM. According to results, for natural pozzolan added cements grinding method affects the microstructure of hydration significantly. For slag added cements no remarkable differences were found between the products of intergrinding and separately grinding from hydration points of view. The gap between the strengths of the cements produced by different methods, decreases with age, providing higher differences at earlier ages.

ÖZET

Bu çal›flmada portland çimentosu klinkeri, do al puzolan ve granüle yüksek f›r›n cürufu kullan›larak beraber ve ayr› ö ütme tekni iyle % 25 katk›l› çimentolar üretilmifltir. Bütün çimentolar (iki adet do al puzolanl›, iki adet granüle yüksek f›r›n cüruflu ve 1 adet portland çimentosu) 3500 ± 100 cm^2/g Blaine inceli indedir. Ayn› kompozisyona sahip, beraber ve ayr› ö ütmeyle üretilmifl çimentolar, hidratasyon mikroyap›s›ve dayan›mlar yönünden 2, 7, 28 ve 90 günlük yafllarda karfl›laflt›r›lm›flt›rBu amaçla belirtilen yafllarda çimento pastalar›nda SEM incelemeleri yap›lm›fl ve çimentolar›n bas›nç dayan›mlar› tespit edilmifltir. Ayr›ca SEM ile çimentolar›n ve mineral katk›lar›n ö ütülmüfl halde flekillerine ve boyutlar›na bak›lm›flt›r. Sonuçlara göre, do al puzolanl› çimentolarda uygulanan ö ütme tekni i hidratasyonu önemli mertebelerde etkilemektedir. Cüruflu çimentolarda ö ütme tekni inin önemli bir etkisi görülememifltir. Ayn› kompozisyonda ve farkl› ö ütme teknikleriyle üretilmifl çimentolar aras›ndaki dayan›m fark›n›nzamanla azald› › tespit edilmifltir.

Modernization of Sample Preparation at Lone Star Industries Greencastle, Indiana

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Abstract

In 1994 a Master Plan was developed to upgrade the control system at Greencastle. The plant was first built in 1969 and systems in use reflected technologies of that time. A central part of this Master Plan had to be an upgrade of the automated sampling and sample preparation line that was installed in 1979 and was quite innovative for that time.

The sampling and sample preparation line in existence since 1979 was set up to accept wet slurry from a raw mill, dry it, and then pass it through an automatic pulverizing mill and automatic pelletizing press. Pressed pellets were then transferred to a Philips PW1660 x-ray fluorescence spectrometer for analysis.

In 1998 a new line was installed that used a new automatic fusion machine as its centerpiece to allow the laboratory to produce fused beads for XRF analysis in the place of the pressed pellets.

EFFECT OF STEAM INJECTION ON MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF CEMENT AND BLAST FURNACE SLAG

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1. ABSTRACT

The isolation of zones is a critical issue in the cement slurry design for steam injection wells. This paper presents the effect of the steam injection process on mechanical and morphological properties of hardened Portland cement and Blast Furnace Slag. Changes in the dimensional stability, permeability and thermal conductivity are also reported.

Four systems of slurries were investigated: one was conventional Portland cement as a reference and three other systems contained blast furnace slag as the cementitious material each using a different drilling fluid as the base. The phase composition, morphology and microstructure of the formed hydration products are reported. The results of the physicochemical properties were related to the values of compressive strength, dimensional changes, permeability and thermal conductivity of the hardened cement. The results show that blast furnace slag slurries provide more temperature stability and less permeability than Portland cement ones. These facts indicate that the blast furnace slag slurries are more appropriate cementing materials for steam injection wells.