ABSTRACTS

THIRTY-SEVENTH INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY



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SEATTLE, WASHINGTON, U.S.A. May 03 – 07, 2015

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VALIDATION OF AN AUTOMATED SCANNING ELECTRON MICROSCOPY (SEM) TECHNIQUE FOR THE CHARACTERIZATION OF CEMENTS

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ABSTRACT

Modeling the hydration of Portland cement based materials using the VCCTL requires detailed characterization of the cement of interest, a process that involves a combination of backscatter Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray spectroscopy (EDX). The data obtained via these methods are merged to produce a segmented phase map from which phase specific volume and surface area fractions are measured. Typically, at least one dozen image fields are acquired to obtain representative measurements for an individual cement. Using a thermal emission, manually controlled scanning electron microscope to implement this process is time intensive, and the manual combination of backscatter images and EDX elemental maps is laborious and prone to error. An automated method to characterize cements based on these procedures has been developed, using a computer controlled SEM (CCSEM) in conjunction with automated image segmentation routines. The robustness of this automated method has already been demonstrated, but a direct comparison to the original method using standard reference materials has yet to be performed. The following paper presents an evaluation of the accuracy and repeatability of the automated method compared to the original manual method using a standardized reference cement, combined with analysis of the same image fields on the same sample using the two techniques.

KILN FEED BURNABILITY REEXAMINED

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ABSTRACT

Burnability of kiln feed is referenced in several publications. Specifics of the methods are described and are used for comparisons between samples; typically one kiln feed currently in use and one with new materials or processes. The burnability index is based on calculations that use particle size including percent quartz >45um and percent limestone >125um counted microscopically and elemental analysis. If the material is burned in a high temperature furnace the free lime is measured by wet methods, mostly following the Franke method. With advances in computer controlled furnaces, automated acid titration, x-ray diffraction with Rietveld refinement, laser particle sizing, and x-ray fluorescence a more refined approach can be established. Additionally, by using a standard reference kiln feed made using reagent grade materials, comparisons can be made between samples from any source and time period. With today's combination of a wide range of raw material choices which may include by-products of other industries and the "Green" movement which is environmentally responsible and resource efficient minimizing waste in any process, the use of advanced techniques is desirable. This paper will describe the techniques used to evaluate differences in four kiln feeds from different US source regions.

APPLICATION OF ALKALINE EARTH CHLOROACETATES FOR THE SETTING CONTROL OF CALCIUM ALUMINATE CEMENTS

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ABSTRACT

Organic and inorganic additives are used to control the setting behaviour of calcium aluminate cements (CAC). As LiCl and Li_2CO_3 typically act as accelerators [1,2], organic acids like citric, formic, benzoic, naphthoic, glycolic, oxalic or tartaric acid and their alkaline earth salts retard the hydration reaction depending on the concentration used[1-9]. In a detailed report on the hydration behaviour of a CAC using Ca-salts of chlorinated acetic acids, calcium-monochloroacetate was pointed out as the strongest retarder [6]. However, in an alkaline ambient a hydrolysis of monochloroacetate into glycolic acid and Cl⁻ takes place [10]:

 $ClCH_2COOA + H_2O \leftrightarrow ClCH_2COOH + A^+ + OH^- \rightarrow HOCH_2COOH + A^+ + Cl^-$

For Li and Mg this conversion reaction is not complete and addition compounds Li(ClCH₂COO)(HOCH₂COOH) or Mg(HOCH₂COO)(ClCH₂COO) are formed. The Ca, Sr and Ba mono-, di- and trichloroacetates (MCA, DCA and TCA) were found to be more stable. However, for the phase identification by XRD appropriate database entries are lacking. Crystal structural data are available for NH₄-, Li-, Na-, Ca-, Ag-, Ni-monochloroacetates/-hydrates [11-17].

In this study crystal structural data of the different alkaline earth chloroacetates and hydrates necessary for the phase identification are presented. All these compounds crystallised from aqueous solution form layered structures with the chloromethyl groups roughly directed in stacking direction on both outsides of the layers. The interlayer distances depend on the cationic radius of the alkaline earth and on the number of substituted Cl. By thermal analysis the anhydrous phases are found to be stable at least up to 200 °C. The retarding effect on the setting of CAC as reported by [6] is additionally influenced by the alkaline earth. As an example Fig. 1 demonstrates an increasing delay of the onset for Mg < Ba < Sr < Ca. In addition to that the pore solutions of the reacted mixtures are chemically analysed to determine the different release rates of Cl⁻ for mono-, di- and trichloroacetates used as admixtures.



Fig. 1. Isoperibolic heat flow calorimetry of CAC (Secar51) with different alkaline earth monochloroacetates used as admixture.

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XRD WITH CLUSTER ANALYSIS ON THE EVALUATION OF PORTLAND CEMENT WITH ADDITIONS

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ABSTRACT

The aim of this work is the study of materials that can be used as additives in the cement industry by X-ray diffraction with cluster analysis. This technique is performed using a group of statistical methods that identify groups of samples with similar characteristics.

The cluster analysis does not require any specific knowledge to interpret the diffactograms and classify the different kinds of additives used in the cement industry. The technique can be applied to the prospection of different types of limestones (calcitic, dolomitic and siliceous) and also to the qualification of different clinkers.

Data presented by the SNIC (Sindicato Nacional da Indústria do Cimento) shows that the Brazilian cement industry produced 64 million ton of cement in 2012, with higher contribution of cements CP-II (slag), CP-III (blast furnace) and CP-IV (pozzolanic). The industrial byproducts more often used in the Brazilian cement industry are the calcined clays, fly ashes and blast furnace slag.

If there is a global environmental interest to produce cement with high quality byproducts, the cluster analysis presents itself as a fast and efficient technique to study and cluster materials with pozzolanic activity.

The samples in this study have different origins: fly ashes comes from different power stations from the South region of Brazil, slag comes from different steel works plants from the Southeast region. Cements with different additions of limestone and white Portland cement were also used.

QUANTITATIVE X-RAY POWDER DIFFRACTION ANALYSIS OF PORTLAND CEMENTS: PROFICIENCY TESTING FOR LABORATORY ASSESSMENT

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ABSTRACT

The current ASTM standard test method for powder diffraction analysis of cements provides guidance, but not an explicit method, for quantifying phase concentrations. The standard utilizes qualification criteria, where an analysis of a set of certified reference materials must fall within stated precision and bias limits. Validation of X-ray powder diffraction analyses by the Rietveld method is particularly important because the normalization inherent in the mass fraction calculations can obscure bias. Currently, the only certified reference materials for phase abundance are a set of NIST SRM clinkers.

A set of portland cements was distributed to 29 laboratories for analysis according to each lab's individual protocols. To provide each lab with quantitative feedback on its precision and accuracy, results are presented graphically with Youden plots. These plots incorporate a ranking to illustrate relative lab precision and accuracy based upon a consensus mean for each phase and ASTM C1365 performance qualification criteria. Labs that fall outside of the compliance limits are provided with information via the plots to assess their systematic and random error. Proficiency testing of this sort provides laboratories with a quantitative assessment of their performance relative to peers using a wider range of materials encompassing the broad spectrum of modern hydraulic cement production. Such a quantitative assessment could be used to qualify laboratories and may be stipulated in a specification.



Figure 1. Youden plot of three replicate X-ray powder diffraction determinations of two different cements provides insight on lab protocol precision and bias.

RIETVELD REFINEMENT INTERPRETATION AND PHASE COMPOSITION OF CLINKER AND PORTLAND CEMENT USING X-RAY DIFFRACTION DATA

David E. Simon, Ph.D.¹

ABSTRACT

Over the past 20 to 25 years the use of Rietveld refinement interpretations has become more and more common in the cement industry. Initially, cement and clinker phase abundance was estimated from the elemental composition determination, i.e., Bogue calculation. However, through use of Rietveld refinement interpretation, cement and clinker phase composition can be estimated directly from X-ray diffraction data.

The procedure used is dependent on the desired outcome, detailed composition of all phases detected or quality control based on the major phases – Clinkers ---C3S, C2S, C3A and C4AF and the calcium sulfate contents of Portland cement.

The detailed composition determination involves one or more extractions, salicylic or maleic acid to remove the C3S, C2S, free lime and portlandite phases, followed by an ammonium chloride extraction for removal of the sulfate phases. These extractions are reproducible and allow for detection and determination of the trace to minor phases including the alkali sulfates, and alkali calcium aluminates and fly ash.

For quality control purposes, Rietveld refinement of only the clinker and Portland cement allow for trends in composition to be continuously monitored and corrective actions taken when the trend varies outside established limits.

Finally, measurement of free lime by X-ray diffraction data analysis is used for quality control purposes in cement plants. This technique is very rapid, and used by technicians to screen the amount of free lime in the clinker within set limits of concentration. This eliminates the need of running the glycol extraction for free lime on every control sample, and the extraction only performed on samples indicated to be outside of the set limits.

¹DES Consulting

QUANTITATIVE RIETVELD PHASE ANALYSIS IN CEMENT: ADVANTAGES AND PITFALLS

Dr. Prof. Gilberto Artioli - Univ. of Padua, Padova, Italy

ABSTRACT

The full profile analysis of complex materials such as cement and binders is a powerful technique to overcome the problems intrinsically connected with conventional QPA methods, such as RIR, single calibration, enf flushing methods. However, the refinement may suffer from a number of pitfalls, such as improper structure models (especially for poorly crystalline phases such as CSH), strong peak overlap, phase pseudosymmetry, and many more. Furthermore the ubiquital presence of one or more amorphous phases needs rescaling of the QPA perfomed on the crystalline components. This is commonly performed using internal standards, either artificially added to the mixtrure or already present in the specimen (G factor technique). The different refinement strategies will be discussed with reference to speficic case studies.

PETROGRAPHIC EVALUATION OF CONCRETES FROM DAISAN KAIHO (SEA FORT NO.3) AT TOKYO BAY, DEMOLISHED ABOUT 80 YEARS AFTER COMPLETION

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Taiheiyo Consultant Co.Ltd

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ABSTRACT

Sea-dredged concretes of an artificial island, which had disintegrated at the coastal revetment due to the Great Kanto Earthquake in 1923 two years after its completion and had subsequently sunk into the sea due to waves, were examined petrographically. Steelreinforced concrete caisson (wall and infill) of a breakwater built in the 1910s and nonreinforced concrete of a warehouse (wall) contained cement particles showing characteristic of the production by the rotary kiln. Belite predominated forming a large cluster composed of small crystals often with a pale colored cross-lamellae, while alite was subordinate occurring in separate particles. Occasionally, slender alkali-aluminate was associated interstitially. These suggest that the coarsely grained raw meal was poorly to moderately burned, resulting in a highly heterogeneous clinker with varying cooling effects on quenching. The gravel used in the reinforced caisson was similar in size to that in the modern concretes, but it was much coarser in the non-reinforced members, suggestive of a size specification of the aggregate for the usage. Chert and sandstone pebbles in the warehouse concrete produced microscopic cracks with surrounding ASR gel. Characteristics of the cement hydrates and the behavior of the chloride ions in concrete were discussed reviewing previous data. These observations revealed the early stage of the rotary kiln 100 years ago that had replaced the bottle kiln in the cement industry in Japan.

竣工後約80年で解体された東京湾第三海堡のコンクリートの岩石学的評価

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太平洋コンサルタント

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竣工2年後に発生した関東大震災

(1923年)により護岸が崩壊し、その後の波浪により海没した人工島の海中より引揚げられたコンクリ ート試料を岩石学的に調査した。防波堤として1910年代に造られた鉄筋コンクリートケーソン(壁体 ・中詰め)と無筋の倉庫のコンクリート(壁体)中に残存するセメント粒子は、回転窯焼成による製 造の特徴を有していた。ビーライトは優勢で細粒の結晶より成る大型のクラスターを形成し、しばし ば淡色でクロスラメラを有するが、エーライトはこれに次ぎ別の粒子中に含まれている。間隙質には ときに細いアルカリアルミネートが発達する。以上より、粗い原料が不十分ないし中程度に焼成され て不均一なクリンカー組織を形成し、さらに冷却時に種々の冷却効果を生じたことを示唆している。 粗骨材の砂利の粒径は、鉄筋コンクリートケーソン中のものは現在と同程度であるが、無筋コンクリ ート中のものは粗いことから、用途による骨材粒度の規定があったことを示唆する。倉庫のコンクリ ートには、骨材中のチャートと砂岩の礫には顕微鏡的なひび割れとASRゲルが生成していた。このほ かに、コンクリート中のセメント水和物の特徴と塩化物イオンの挙動についても、過去のデータを引 用し、検討を行った。以上の観察から、徳利窯から回転窯に転換後の、100年前のわが国の初期のセ メント製造技術の一端を伺い知ることができる。

MICROSCOPIC CHARACTERIZATION OF CRYSTALLINE AND AMORPHOUS VARIETIES OF SiO₂ EXHIBITING DIFFERENT ASR POTENTIAL

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ABSTRACT

Both crystalline and amorphous varieties of SiO_2 from several localities in the Czech Republic and Sweden were subjected to experimental testing by use of accelerated mortar bar test (AMBT, following ASTM C1260), and various microscopic techniques (polarizing microscopy combined with cathodoluminescence (CL) microscopy, spectroscopy, and petrographic image analysis). The expansion values of the mortar bars were correlated with phase and with the microstructural and CL spectral characteristics of selected aggregates.

The highest expansion values (0.534%) were indicated by very fine-grained chert (mean equivalent diameter, EqD_{mean}, of 0.013 mm), containing significant amounts of fine-grained to cryptocrystalline matrix. The chert exhibited a dark red CL emission band at about 640 nm with a low intensity. The medium expansion values (0.151-0.282%) were indicated by fine-grained orthoquartzite (EqD_{mean} of 0.092-0.205 mm), as well as metamorphic vein quartz separated from phyllite (EqD_{mean} of 0.060 mm). The orthoquartzites showed various CL of different quartz grains, from blue (with growth zoning in some cases) through violet, red, and brown. Two CL spectral bands at ~450 and ~630 nm, with various intensities, were detected. The CL characteristics of red as well as zoned blue CL quartz grains are typical of those from a volcanic source. The quartz from phyllite displayed an inhomogeneous dark red CL with two CL spectral bands of low intensities at ~460 and ~640 nm. The lowest expansion value (0.085%) was indicated by massive coarse-grained pegmatite quartz, displaying a typical short-lived blue CL with rare secondary fluid trails with a darker CL (~480 nm).

Of the rocks mentioned above, the expansion seems to be closely associated with grain size as well as with the presence of a SiO_2 -rich very fine-grained to cryptocrystalline matrix. Even a small volume of the matrix (4-10 vol.%) increased the expansion values into the area of

reactive samples (expansion > 0.100%). The role of quartz deformation was principally obvious in the case of quartz from pegmatite (resp. quartz from phyllite), displaying undulose extinction (resp. characteristics typical of low-to-medium temperature recrystallization mechanisms of bulging and subgrain rotation). It is more complicated to interpret the ASR potential of hydrothermal vein quartz (open space filling) related to Pb-Zn-(Ag) mineralization. The hydrothermal quartz contained fine-grained quartz grains (EqD_{mean} of 0.117 mm) and no cryptocrystalline matrix. In spite of this, the expansion value reached 0.132% after 14 days of AMBT treatment. The higher ASR potential of the studied hydrothermal quartz may be connected with high concentrations of defect centers, and probably with amorphized micro-regions in the quartz, respectively; indicated by an unstable yellow CL emission (~570 nm).

THAUMASITE SULFATE ATTACK: CASE STUDIES AND IMPLICATIONS

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ABSTRACT

The thaumasite form of sulfate attack (TSA) is a unique distress mechanism in portland cement concrete in which thaumasite formation (TF) alters the primary binder, calcium silicate hydrate (CSH), in addition to calcium hydroxide and calcium aluminate hydrates. TSA in concrete may cause loss of paste-aggregate bond, strength, coherence, and eventually serviceability. The time frame of TSA can be as short as a couple of years. Reported TSA cases have mostly involved sulfate from external sources. This paper presents two less common TSA cases, in which sulfate was determined to be from an internal source: in one case, from dolostone coarse aggregate and in the second case from the cementing material. Characteristics of TSA distress and the composition and texture of the concrete are discussed, and these cases are compared with other reported internal and external TSA cases. It is concluded that petrographic examination of the concrete, particularly using thin-sections, provides the most definitive diagnosis of TSA. Significant implications of findings from the two case studies regarding mechanisms of thaumasite formation and potential propensity of portland-limestone cement for TSA are also discussed.

KEYWORDS: Thaumasite sulfate attack (TSA), concrete durability, concrete deterioration, limestone, dolomite, concrete petrography, gypsum



DETERMINATION OF WATER TO CEMENTITIOUS (W/CM) BINDER RATIOS BY THE USE OF THE FLUORESCENT MICROSCOPY TECHNIQUE IN HARDENED CONCRETE SAMPLES: PART IV.

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ABSTRACT

The relationship between the water to cement (w/c) ratio of a cement-based matrix and its capillary porosity, and by association the intensity of fluorescent light developed in a particular sample due to impregnation with a fluorescence resin, is well documented. It is also well reported that for 28-day old Portland cement-only concrete mixes hydrated under normal conditions this relationship is valid for w/c ratio assessments between 0.35 and 0.70. Earlier parts of this study confirmed similar relationships exist with various supplementary cementitious materials at differing w/cm ratios. However, it also showed a consistently higher fluorescence developed with the various SCM-containing mixes when compared to the equivalent Portland cement-only references. Either there truly is greater pore space available for the fluorescent epoxy to fill due to delayed hydration/ pozzolanic reaction, or the inherent light transmission characteristics of the SCM's allow light that is normally blocked by nonhydrated cement particles to shine through, or both. The purpose of this work was to test non reactive fillers of different light transmission properties to help resolve the question of inherent light transmission, and to evaluate later age specimens to see if lower levels of reaction at 28 days are partially responsible for the brighter images seen developing with SCM's.

The results presented indicate that the greater the amount of various SCM's used, the greater fluorescence light intensity developed compared to the Portland cement only equivalent mixes. All the material mixes tested showed a broad gradient relationship between the fluorescence light intensity and the w/cm ratio. The light intensity of the various SCM mixes appears to relate to the relative amounts of SCM material and to the material's apparent translucency. Testing of mixes containing similar replacement levels of various inert fillers confirm that the transparency of the original filler material does affect the fluorescence light

intensity of the resulting cementitious paste. Specifically, the mixes containing the more transparent filler showed a higher fluorescence light level than the mixes containing the more opaque filler, which apparently masked the transmitted fluorescent light.

Finally, this paper describes the findings from sets of companion test samples produced at 18 months of age. The results show little difference in the degree of fluorescence developed by the mixes containing 100% Portland cement, 25% C-ash, 25% F-ash and 7.5% microsilica when compared to the 28 day old samples. The sample sets containing fly ash and microsilica had generally slightly lower light intensities suggesting some additional slight hydration and activation has occurred. The 50% slag samples, however, continued to show the high levels of fluorescence at higher w/cm ratios. Additionally, the 18 month old samples containing SCM's still exhibit a greater fluorescent light intensity than the equivalent Portland cement samples. This underscores the absolute requirement for appropriate reference samples when applying this technique to mixes containing SCM's.

FROM QUARRY TO STRENGTHS: HOW COMPOSITION OF RAW MEAL AFFECTS CLINKER QUALITY AND CEMENT ADDITIVES FORMULATION

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ABSTRACT

The effect of chemical and mineralogical composition of raw materials, including the influence of minor elements, on clinker and cement quality has already been widely discussed in literature since the discovery of Portland cement. From the beginning of the raw meal preparation to the choice of the right chemical additive, modern cements require a global approach to optimization, in which microscopy techniques always play a key role. In this paper we present a detailed study of cement performances, taking into account several clinkers and related raw meals, kiln feeds and quarry materials. In each case, the most suitable cement additive formulation will be discussed, with the target to improve overall performances during cement manufacturing and use.

INFLUENCE OF CURING ON SULFATE ATTACK OF CEMENT-LIMESTONE BASED PASTE AT LOW TEMPERATURE

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ABSTRACT

Sulfate attack is one of the most important factors that influences the durability of concrete structures. Extensive researches have been carried out on the conventional sulfate attack, while it has been found that the thaumasite form of sulfate attack (TSA), sulfate attack at low temperature, has just been discovered and its mechanism is not well understood so far. In this study, the sulfate attack of cement paste incorporating 30% mass of limestone powder was investigated. After 20 °C water cured for 7 days, 14 days and 28 days respectively, 20 mm cube specimens were exposed in a 5% magnesium sulfate solution at (6 \pm 1) °C for periods up to 240 days. Their appearance change, compressive strength development were examined at different storage time, and selected paste samples were examined by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and energy dispersive spectrum (EDS). The results indicate that all Portland-limstone cement pastes suffer from appearance deterioration to some extent. The compressive strength of cement paste initially increases and after 120 days decreases with the exposed period. In addition, the cement paste with short curing time is more susceptible to sulfate attack, which directly leads to the formation of non-binder thaumasite crystal accompanied with the formation of ettringite, gypsum and brucite, and becomes a white, mushy, and incohesive matrix. Additionally, the extent of sulfate attack is greater and the formation of thaumasite is observed earlier for shorter curing time.

Key words: Thaumasite; Sulfate attack; Limestone; Curing; Low temperature

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INVESTIGATION OF THE INTERFACIAL TRANSITION ZONE OF LIGHTWEIGHT AGGREGATE AND RICE HUSK ASH USED AS PHASES CHANGE MATERIAL CARRIERS

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ABSTRACT

Using Phase Change Materials (PCMs) in buildings decreases the energy consumption of the HVAC system and increases occupant comfort. As PCM cannot be added to cementitious media directly, different carriers have been proposed to indirectly incorporate PCM in building materials. Because of their porous structures, Lightweight Aggregate (LWA) and Rice Husk Ash (RHA) can both absorb and contain PCM, although it is very likely that a portion of the PCM sticks to the surface of LWA and RHA and subsequently affects the chemical, physical, and mechanical properties of the media. The object of this study is to investigate the Interfacial Transition Zone (ITZ) of PCM-presoaked LWA and RHA when introduced in cementitious matrices. Two microscopic methods, Scanning Electron Microscopy (SEM) and microscopy-assisted Fourier Transform Infrared Spectroscopy (FTIR), were used to investigate the structure and the composition of the ITZ and weaken the contact between the carriers and the cementitious matrix.

Keywords:

Interfacial Transition Zone (ITZ), Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR).

MICROSTRUCTURAL CHARACTERISATIONOFWELLBORE CEMENT SHEATHUNDER HIGH TEMPERATURE AND PRESSURE

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Key words: Wellbore Cements, TEM, Microstructure

ABSTRACT

Wellbore cement sheath provides zonal isolation between the casing and formation to prevent gas migration to the surface. Fractures in the cement sheath and along the cement boundaries will lead to pollution of the environment. Fractures within the cement sheath typically result from the impact of pressure on the cement during hydraulic fracturing operations and degradation of cement at high temperatures. The physical and chemical behavior of wellbore cement significantly changes at elevated temperatures in geothermal and thermal recovery wells. In HT/HP conditions, CSH gel is changing to metamorphosis, which usually results in decreased compressive strength and increased permeability at high temperature. The microstructure of cement hydration is therefore essential at high temperatures and pressures.

In this study, the Transmission Electron Microscopy (TEM)was used to investigate the morphology of hydration products before and after the thermal loading. TEM shows that there is degradation of ettringite at high temperature. The CSH gel is more amorphous and dense compared to normal CSH structure. There is also about 5% shrinkage of CSH gel after the thermal loading because of CSH gel is convert to alpha dicalcium silicate hydrate. The porosity and permeability of cement is increasing at high temperature while the compressive stress decreases, which corresponds to the observation of TEM of cement hydration products.

INVESTIGATION OF SELF-HEALING REBAR COATINGS FOR REINFORCED CONCRETE

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ABSTRACT

The more than \$100 billion spent annually in the U.S. on infrastructure maintenance is not enough to keep the built environment in a state of good repair. More funding, however, is only a temporary solution – it is widely acknowledged that new materials, with better durability, are the key to addressing this significant challenge. One of the most popular methods of delaying the onset of electrochemical corrosion in reinforced concrete structures is to use epoxy-coated rebar (ECR). Chips or cracks in the epoxy coating, which can be easily introduced during handling or at the work site, significantly reduce the effectiveness of this technique.

This research focuses on the development of self-healing rebar coatings containing encapsulated tung oil. Microscopy was used to characterize the self-healing coatings, as well as the interfacial transition zone between the coatings and the cement paste. The healing ability has been characterized by accelerated corrosion testing. The impact of self-healing coatings on steel-reinforced concrete mechanical properties has also been evaluated. The corrosion tests have been run on large-scale specimens that can easily be scaled up further.

INVESTIGATION OF FLY ASH ACTIVATION WITH CHEMICAL ADDITIVES: INTERACTIONS WITH PORTLAND CEMENT HYDRATION

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ABSTRACT

The reduction of greenhouse gases emission is the main issue the cement industry is facing now and will be facing in the near future. In the light of this, the use of the so-called secondary cementitous materials is mandatory for all modern cements. Among other types of mineral additions, fly ashes represent one of the most promising, due to availability and hydraulic behavior. In this paper we discuss the reactivity of several fly ash sources in a typical blended cement system, with particular reference to chemical additive used as performance enhancer. Physico-mechanical parameters such as compressive strengths and physico-chemical measurement are discussed and commented, with the aid of microscopy techniques for microstructure evaluation.

BACTERIA MAKE CONCRETE SELF-HEALING: DEVELOPMENT AND APPLICATION OF BACTERIA-BASED SELF-HEALING CONCRETE

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ABSTRACT

In this research project the suitability of very specific but otherwise harmless bacteria are tested for their ability to repair cracks and thus significantly improve the durability of concrete structures. Such a bacterial repair mechanism would be beneficial for the economy and the environment at the same time, as concrete is worldwide the most applied building material. This new type of 'bio-concrete' would make costly manual repair unnecessary and would minimize the use of raw materials, as structures will last much longer.

In nature a huge number of different varieties of bacteria occur and some of these are likely well adapted to artificial man-made environments such as concrete. From a human perspective concrete may seem an extreme environment as the material is dry and rock-solid. However, this does not apply to a specialized group of bacteria, the 'extremophiles', named after their habit to love extreme conditions. Some of these bacterial species are not only known to love extremely dry conditions, but also to be able to produce copious amounts of limestone. This calcium carbonate-based material, as well as other types of bio-minerals produced by bacteria, could serve to seal or heal cracks in concrete.

The functionality of a number of developed 'healing agents', comprising encapsulated bacterial spores and nutrients, has been confirmed in both experimental laboratory studies and specific outdoors applications. Three products are currently being developed and tested in practical applications, 1. Self-healing concrete, 2. Self-healing repair mortar, and 3. A sprayable liquid repair system.

EFFECT OF CALCIUM SOURCE ON MICROBIAL SELF-HEALING OF CONCRETE

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ABSTRACT

Brittle construction material such as concrete cracks due to excessive loading or exposure condition and leads to deterioration and loss of structural integrity over time. Concrete structures are constantly in need for repair which is a costly and labor intensive process. Recent research suggests that natural cementation from microbial calcium carbonate precipitation can heal cracks in concrete and hence, can provide an economical alternative to traditional repair methods. In this study, role of various calcium sources such as calcium chloride, cement paste powder and calcium lactate was investigated to supply calcium ions for the bacteria Sporosarcina pasteurii to produce calcium carbonate deposits. The motivation for the study is to determine if internal supply of calcium ions through partial dissolution of calcium ion bearing phases in concrete such as remaining unhydrated cement particles, calcium hydroxide and calcium silicate hydrate are sufficient or if externally supply is needed to facilitate biomineralization in concrete. An array of characterization techniques namely Xray diffraction, thermal analysis and scanning electron microscopy were used to characterize and compare calcium carbonate deposit obtained through use of different calcium sources. In addition, cracked mortar beams were treated using bacteria and healing efficiency was measured through resonance frequency and crack tip opening displacement controled three point bending test.

SELF-REPAIRING CONCRETE: APPROACHES AND CHALLENGES TO BIO-BASED CONCRETE REMEDIATION

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ABSTRACT

Biogenic calcium carbonate precipitation by bacteria has been identified as a novel method to improve durability and remediate cracks in concrete. This presentation will discuss some of the approaches to incorporating bacteria into concrete as well as the challenges and advantage of each method. An overview on some of the research activities occurring at UT Austin regarding utilizing bacterial agents to improve the properties of concrete will also be given.

DEVELOPMENT OF RELATIONSHIP BETWEEN CLINKER PHASE COMPOSITION, POROSITY AND BOND WORK INDEX

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ABSTRACT

Portland cement is a conglomerate of minerals generated during burning process and solidified by the liquid phase. One important property is the resistance of the clinker to mechanical loads i.e. its grindability. It is well known that resistance to mechanical stress and the grindability of clinker has a considerable influence on the operation of grinding equipment, on the energy requirement of grinding, affects the Particle Size Distribution and quality of the cement produced. The mechanism of clinker grindability and the relation between the grindability and the burning condition of clinker are extremely complicated. It seems impossible to make a theory which can be applied to the plant operation. Grindability of clinker principally depends on the chemical composition, microstructure, mineralogy of clinker and grinding method. It is experimentally known, that the grindability strongly changes according to the burning conditions. The aim of this investigation was to determine clinker chemico-mineralogical and micro-structural parameters which fundamentally influence grindability. In the study, the grindability and microstructure of 18 commercial clinkers (C1 to C18) having different chemico-mineralogical composition have been investigated. The study attempts to develop a linear & non-linear mathematical correlation between the Bond's work index and chemico-mineralogical and micro-structural critical parameters.

Key words: Microstructure; Bond Index; clinker; OM

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PETROGRAPHIC EXAMINATION OF MORTAR-LIKE SAMPLE FROM SITE NEAR ANCIENT RUINS OF QUMRAN, ISRAEL

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ABSTRACT

This study reports mineralogical and petrographic characterization of a mortar-like sample from a site near the ancient ruins of Qumran, Israel, where the Dead Sea Scrolls were discovered. The Dead Sea Scrolls, dating back to 200 B.C., were found in a series of caves around the site between 1946 and 1956. The sample was taken from an odd shelf-like structure found at a suspected cave entrance at the site during a recent excavation. The sample has been studied by means of petrography and SEM-EDX in order to determine whether the strange formation was "natural" or man-made.

Petrographic examination reveals that the sample consists of sand in a buff-gray calcareous binding matrix. The sand consists mainly of sub-rounded to sub-angular particles of limestone, dolomitic limestone, and argillaceous limestone with lesser amounts of chert and glassy materials (volcanic glass). Despite the abundant recrystallization and replacement of the matrix by secondary aragonite, patches of fine-textured original cementing paste still remain. Some ghost or relict particles observed in this fine-textured matrix exhibit optical and elemental composition consistent with calcined limestone or carbonated lime, suggesting a man-made material.

This study demonstrates that petrography is a valuable tool for the evaluation of ancient mortars. In addition to the mortar constituents (aggregates and binder), petrography provides information on the secondary products (recrystallization) and the extent of alteration during the course of aging.

POROSITY MEASUREMENT OF HYDRATED OIL WELL CEMENTS

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ABSTRACT

Electrical resistivity is a recent technique that is more widely accepted for determining porosity in cementitious systems as it has proven to be robust and non-destructive. In this work electrical resistivity was determined on three oil well cementing systems having density 1901 kgm⁻³, 1876 kgm⁻³ and 1200 kgm⁻³ or w/c ratios of 0.44, 0.59 and 1.80. These were considered as a normal cement, thermal cement and lightweight cement respectively. Two geometries were investigated, uniaxial and embedded rod and compared and in the present investigation the embedded rod geometry was determined to be the most appropriate. Electrical resistivities using the embedded rod geometry was found to be consistent with porosity obtained by BSE and image analysis, He pycnometry and Hg Porosimetry for the normal and thermal hydrated cements. The lightweight cement showed a different trend in electrical resistivity and porosity measurements that related to the higher water content, faster hydration and pozzolanic reactions. These correlated well with BSE images and imaging. As such the electrical resistivity of the hydrated cement was shown to be a function of its microstructure, in terms of the porosity, degree of chemical reaction and on the chemistry of the pore solution based on published data.