PROGRAM INTRODUCTION

It is my great pleasure to welcome all participants to the 39th meeting of the International Cement Microscopy Association in Toronto/Canada.

Toronto and the Crown Plaza Toronto Airport Hotel do host the 39th meeting of ICMA (WEBSITE: WWW.CEMMICRO.ORG) from 9.4.2017 – 13.4.2017. Again an interesting and variable program of presentations and panel discussions will be provided and visits to selected sites are included. Among others visits to the SHAWCOR manufacturers and HUMBER ARBORETUM are included.

ON Thursday a visit to the world famous Niagara Falls completes the 39th ICMA meeting in 2017. The presentations and manuscripts are collected and are available as proceedings of the 39th ICMA meeting in Toronto (ISBN: 1-930787-16-2). Also the number of reviewed papers increased from last year to this year again. The classic manuscripts also take part and still make the main part of the proceedings. Both types of manuscripts are warmly welcome for the proceedings.

For the future many reviewed papers should be included in these proceedings, but still reports on different topics on cement concrete and other building and construction materials and also on methods like microscopy and other physical and chemical determinations are warmly welcome.

Many scientists and application experts are coming to the meeting sharing their expertise on all the broad field of building and construction materials from the quarry to the final product including topics as sustainability and long term stability. Environmental and ecologic aspects play a more important role in the field of raw materials and secondary materials.

Highly important for ICMA is the strong inter correlation between industrial research and universities and the broad field of applications. For this the participation of young scientists in the annual ICMA meetings is wanted. Special important topics are always covered by presentations provided by high levelled interesting panel discussions brought together by Dr. Karen Luke.

To complete the whole meeting also a ladies and spouse program for accompanying people will be provided headed (Spouse & Guest Committee: Lourdes M. Nisperos-Chairman; Maria Poellmann & JoAnn Buchanan-Co-Chairmen).

Special thanks are also due to the local organizing committee headed by Dr. Karl Peterson.

I wish all participants a fruitful and enjoyable time in Toronto and a nice visit to Canada.

Dr. Herbert Poellmann (Program Committee Chairman)
PRELIMINARY PROGRAM OF THE THIRTY-NINTH INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY
SPONSORED & ORGANIZED BY INTERNATIONAL CEMENT MICROSCOPY ASSOCIATION (ICMA); WEBSITE: (WWW.CEMMICRO.ORG)
APRIL 9-13, 2017
CROWNE PLAZA TORONTO AIRPORT HOTEL, TORONTO, ONTARIO, CANADA

SUNDAY, APRIL 9, 2017

2:00 – 4:00 PM – REGISTRATION
Crowne Plaza Toronto Airport Hotel
Toronto, Ontario, Canada

6:00 – 8:00 PM – Welcome Reception
Hosted by ICMA & Co-Host: ROAN Industries, Inc.

MONDAY, APRIL 10, 2017

8:00 – 9:00 AM – REGISTRATION
Crowne Plaza Toronto Airport Hotel
Toronto, Ontario, Canada

9:00 – 9:20 AM – Welcome & Opening Address
Dr. Arturo G. Nisperos
ICMA General Chairman

9:20 – 9:30 AM – Introduction of Keynote Speaker
Dr. Karl Peterson
Program Committee, Co-Chairman

9:30 – 10:00 AM – Keynote Address
Dr. Chris Rogers
Keynote Speaker
Chair, Technical Committee on Concrete Materials and Construction
Canadian Standards Association
Mississauga, Ontario, Canada

10:00 – 10:10 AM - Program Introduction
Dr. Herbert Poellmann
Program Committee Chairman
10:10 – 10:40 AM – QUANTIFICATION OF SLAG DISSOLUTION IN SUPERSULFATED CEMENT
Rana Masoudi and R. Doug Hooton
Civil Engineering Department
University of Toronto
Toronto, Ontario, Canada

10:40 – 10:55 AM – BREAK

10:55 – 11:25 AM – QUANTITATIVE ANALYSIS ON THE AMORPHOUS HYDRATED MAGNESIUM CARBONATE IN A CARBONATED REACTIVE MgO-PORTLAND CEMENT SYSTEM BASED ON ENERGY-DISPERSIVE X-RAY SPECTROSCOPY
Runxiao Zhang and Daman K. Panesar
Civil Engineering Department
University of Toronto
Toronto, Ontario, Canada

11:25 – 11:55 AM – POTENTIAL VALUE OF TRACE ELEMENTS IN THE SOURCING OF CEMENTITIOUS MATERIALS
Adrienne Weishaar and Aaron R. Sakulich
Department of Civil and Environmental Engineering
Worcester, Massachusetts, U.S.A.
Remya P.M. Narayanan and Marion H. Emmert
Department of Chemistry & Biochemistry
Worcester Polytechnic Institute
Worcester, Massachusetts, U.S.A.

11:55 – 12:10 PM – Introduction of Exhibitors
Dr. Stella Marusin
Exhibit Committee Chairman

12:10 – 1:00 PM – LUNCH BREAK

1:00 – 1:30 PM – EVALUATION OF THE EFFECT OF OCEAN ON THE HYDRATION OF COMMERCIAL MORTARS
Ruslan Liferovich, Mahir Dham, Kelly Hafkey, David Sobransingh
MAPEI Corporation, Research & Development
Deerfield Beach, Florida, U.S.A.

1:30 – 2:00 PM – EFFECT OF CELLULOSE NANOCRYSTAL (CNC) PARTICLES AS AN ADDITIVE ON THE RHEOLOGICAL BEHAVIOR OF OIL WELL CEMENT PASTE
Mohammad Reza Doustri, Yamin Boluk & Vivek Bindiganavile
University of Alberta,
Alberta, Canada
2:00 – 2:30 PM – **PREVENTION OF ASR IN OIL & GAS WELLBORE CEMENTS COMPRISING SILICA-BASED MICROSPHERES**
D. Albers and M. Radonjic  
Sustainable Energy & Environmental Research (SEER) Labs.  
Craft & Hawkins, Department of Petroleum Engineering  
Louisiana State University  
Baton, Rouge, Louisiana, U.S.A.

2:30 – 3:00 PM – **SCANNING ELECTRON MICROSCOPY EXAMINATION OF ALKALI-SILICA REACTION PRODUCTS IN THE ACCELERATED MORTAR BAR TESTING (ASTM C – 1260)**  
Farideh Golmanaki and R. Doug Hooton  
Civil Engineering Department  
University of Toronto, Ontario, Canada

3:00 – 3:15 PM – **BREAK**

3:15 – 6:30 PM – **PANEL DISCUSSION: ALKALI SILICA REACTIVITY (ASR) – A 2017 PERSPECTIVE**  
Conducted by: Dr. Karen Luke, Workshop Coordinator & Chairman

- **Panelists:**
  1. Dr. Tetsuya Katayama – Taiheiyo Consultant Co., Ltd., Sakura, Japan – “*AN ATTEMPT TO ESTIMATE PAST EXPANSION OF CONCRETE BASED ON PETROGRAPHIC STAGE OF ALKALI SILICA REACTION.*”
  2. Prof. Borge Johannes Wigum – Heidelberg Cement Northern Europe/Norwegian University of Science and Technology (NTNU) – “*CURRENT DEVELOPMENTS IN RESEARCH ON ALKALI AGGREGATE REACTIONS AND RILEM OVERVIEW.*”
  3. Prof. Doug Hooton – Department of Civil Engineering, University of Toronto, Ontario, Canada – “**PREVENTING ASR: WHAT ARE WE MISSING?**”
TUESDAY, APRIL 11, 2017

8:00 – 8:05 AM – REGISTRATION & WELCOME

8:05 – 12:00 Noon – WORKSHOP : CEMENT & CONCRETE MICROSCOPY
Conducted by Dr. Karen Luke, Workshop Coordinator & Chairman

- WORKSHOP PRESENTERS:

1. Dr. Pieter du Toit – Consultant Cement Industry Solutions, Austria – “THE USE OF MICROSCOPIC TECHNIQUES FOR PROCESS CONTROL FROM QUARRY TO CLINKER.”


3. Mr. Peter Laugesen, PELCON Materials Testing ApS, Denmark: “TECHNIQUES IN CONCRETE MICROSCOPY WITH SPECIAL EMPHASIS ON POTENTIAL PITFALLS IN PREPARATION AND INTERPRETATION.”

12:00 – 1:00 PM – LUNCH BREAK

1:00 – 1:30 PM – SETTING CONTROL OF CALCIUM ALUMINATE CEMENT USING SUBSTITUTED ACETIC ACIDS AND THEIR CALCIUM SALTS
Ronny Kaden and Herbert Poellmann
Department of Mineralogy & Geochemistry
University of Halle, Halle (Saale), Germany

1:30 – 2:00 PM – IN-SITU HYDRATION STUDY OF YE’ELIMITE-CONTAINING CEMENT BY PYCHOGRAHIC X-RAY COMPUTED TOMOGRAPHY
Ana Cuesta and Miguel A.G. Aranda
ALBA Synchrotron, Barcelona, Spain
Angeles G. De la Torre and Isabel Santacruz
Department of Inorganic Chemistry, Crystallography & Mineralogy
University of Malaga, Malaga, Spain
Pavel Trtil, Juliop C. da Silva, Ana Diaz, and Mirko Holler
Paul Scherrer Institut, Villigen, Switzerland

2:00 – 2:30 PM – STUDY OF THE EFFECT OF A NOVEL METAL-SILICATE NANODISPERSION ON THE WATER PERMEABILITY OF CONCRETE
V. Russo, G. Ferrari and D. Salvioni  
MAPEI, S.p.A., Milan, Italy  
L. Valentini, and G. Artioli  
University of Padova, Padua, Italy  
Mateja Stefancic  
Slovenian National Building & Civil Engineering Institute  
Department of Materials, Laboratory of Concrete, Stone, and Recycled Materials, Ljubljana, Slovenia

2:30 – 3:00 PM – CLUSTERING OF CEMENTITIOUS PARTICLES IN CONCRETE  
Callie LaFleur and Ted Sibbick  
GCP Technologies  
Cambridge, Massachuesetts, U.S.A.

3:00 – 3:15 PM – BREAK

3:15 – 3:45 PM – RAPID SCREENING METHOD FOR HYDRAULIC ACTIVITY OF POZZOLANIC MATERIALS FROM GUATEMALA USING XRD ANALYSIS  
Luis Velasquez, Roberto Diaz, Carmela Barrientos, Elvis Garcia  
CETEC, Cementos Progreso, SA  
Guatemala City, Guatemala

3:45 – 4:15 PM – PARTICLE SIZE ANALYSES OF FLY ASH SOURCES BY LASER DIFFRACTION AND SECONDARY ELECTRON IMAGING  
Abu Yassar and Daman K. Panesar  
Civil Engineering Department  
University of Toronto, Ontario, Canada

4:15 – 4:45 PM – ANALYZING HEAT FLOW AND SHRINKAGE OF A PORTLAND CEMENT MORTAR BY MULTIVARIATE STATISTICAL EXAMINATION OF XRD DATA  
Torsten Westphal and Thomas Bier  
TU Bergakademie Freiberg  
Institut fuer Keramik, Glas- & Baustofftechnik  
Freiberg, Germany

4:45 – 5:15 PM USING OF BOTTOM ASH AND ALUMINUM ANODIZING SLUDGE TO PRODUCE CALCIUM SULFOALUMINATE BELITE CEMENT  
Eugenio Bastos da Costa, Erich D. Rodriguez, and Ana Paula Kirchheim  
NORIE, Universidade Federal do Rio Grande do Sul (UFRGS)  
Porto Alegre, Brazil.  
Erich D. Rodriguez, Department of Civil Engineering IMED, Passo Fundo, Brazil  
Luciano Gobbo, Panalytical, AMEC, Westborough, Massachusetts, U.S.A.
5:15 – 5:45 PM – RECONSTRUCTION OF CRACK PLANE ORIENTATION FROM 2D SLICES OF CORES FROM REINFORCED CONCRETE PANELS AFTER SHEAR STRESS FAILURE
Ekaterina Ossetchkina, Giorgio Proetos and Karl Peterson
Civil Engineering Department
University of Toronto, Ontario, Canada

WEDNESDAY, APRIL 12, 2017

8:00 – 8:05 AM – REGISTRATION & WELCOME

8:05 – 8:35 AM – IMPACT OF TESTING METHODOLOGY ON THE DAMAGE MECHANISM OF SULFATE ATTACK IN PORTLAND CEMENT CONCRETE
Alireza Dehghan, Barry Qui, and Karl Peterson
Civil Engineering Department
University of Toronto, Ontario, Canada

8:35 – 9:05 AM – MICROSCOPY OF 1860 NATURAL HYDRAULIC CEMENT IN CONCRETE FROM EAST BLOCK PARLIAMENT BUILDING
Karl Peterson and Spencer Higgins
Civil Engineering Department
University of Toronto, Ontario, Canada

9:05 – 9:35 AM – A MICROSCOPY APPROACH TO FLY ASH BLENDED CEMENTS: QUALITY, DURABILITY AND PERFORMANCES
A. Lo Presti and M. Magistri
MAPEI S.p.A. R & D. Central Laboratory, Milan, Italy

9:35 – 10:05 AM – PETROGRAPHIC OBSERVATION AND LONG-DURABILITY OF CONCRETE FROM SEA FORT NO. 2 CONSTRUCTED MORE THAN 100 YEARS AGO
Yoko Ando, Tetsuya Katayma, Tomomi Sato, Shinichi Hirono, Kouzou Mukai, Takatoshi Noguchi
Taiheiyo Consultant Co., Ltd. Sakura, Japan
Port and Airport Office, Kanto Regional Development Bureau, MLIT

10:05 – 10:35 AM THE PICTON ONTARIO EXPOSURE SITE – DAMAGE RATING INDEX ASSESSMENT AFTER EIGHTEEN YEARS OF ASR TESTING
Sandeep Sony, Darren Bond, Luz Polo Buitrago, Bishnu Gautam, Heesun Lee
Karl Peterson, and R. Doug Hooton
Civil Engineering Department
University of Toronto, Ontario, Canada
10:35 – 10:50 AM **BREAK**

10:50 – 11:20 AM – **UNIQUE POPOUTS CAUSED BY CALCINED DOLOMITE**
Hugh (Xiaoqiang) Hou, Ella Shkolnik, Paul Krauss, James Connolly

11:20 – 11:50 AM – **MICROSCOPIC CHARACTERIZATION TO STUDY THE MECHANISM OF BENEFICIAL CO2 UTILIZATION IN READY MIXED CONCRETE PRODUCTION**
Sean Monkman,
CarbonCure Technologies
Dartmouth, Nova Scotia, Canada
Kathryn Grandfield,
Department of Materials Science & Engineering
McMaster University, Hamilton, Ontario, Canada
Greg Dipple,
Department of Earth, Ocean and Atmospheric Sciences
University of British Columbia, Vancouver, BC, Canada
Laila Raki
National Research Council Canada
Ottawa, Ontario, Canada

11:50 – 12:00 Noon – **CLINKER EXCHANGE PROGRAM**
Dr. Hugh Hou, Clinker Sample Exchange Committee Chairman

12:00 – 12:10 PM – **ANNOUNCEMENT OF 2018 ICMA CONFERENCE**
Tom Dealy & Dr. Arianna LoPresti
Site Selection Committee Chairs

12:10 -12:20 PM – **CLOSING REMARKS**
Dr. Arturo G. Nisperos
ICMA General Chairman

12:20 – 1:20 PM – **LUNCH BREAK**

1:20 – 7:00 PM – **TOUR OF SELECTED INTERESTING SITES IN METRO TORONTO:**
Coordinated by Dr. Karl Peterson, Program Committee Co-Chair

- 1:20 PM – Depart from Crowne Plaza Toronto Airport Hotel to **SHAWCOR**- Manufacturer of offshore pipelines (with concrete linings). They have an impressive and massive sub-sea simulator for testing pipe with concrete insulator.
• 2:20 PM – Depart Shawcor to HUMBER ARBORETUM – Consists of botanical gardens and natural areas surrounding Humber River. This unique site is home to the Carolinian bioregion, the most diverse ecosystem in Canada and boasts over 1,700 species of plants and animals. The Humber Arboretum covers approximately 250 acres (101 hectares). It was started in 1977 through the initiative of Humber College for the purpose of facilitating research and education, establish and maintain living plant collections, promote conservation and restoration practices and provide a quality visitor experience. The Arboretum includes six kilometers of walking trails with a walking time of approximately 2 hours. Come and explore a world of beauty.

4:00 – 6:00 PM. – ICMA HOSTED RECEPTION AT HUMBER ARBORETUM

• 6:00 PM – Depart Humber Arboretum to BAPS HINDU TEMPLE – Magnificent white limestone structure with concrete – lines temple designed by Professor P. Kumar from University of California, -Berkeley. The BAPS Shri Swaminarayan Mandir Complex consists of: (a) Haveli Tour, (b) Video Presentation, (c) Mandir Tour, (d) Exhibition Tour (optional), (e) Abhishek Mandap (Optional) (f) Gift Shop. Depending upon the availability of time, ICMA tour/trip to BAPS Hindu Temple might not include all the above-mentioned tours. (Note: The Mandir is a house of God. Being a very sacred place, all visitors are required to abide by the following rules and regulations applicable to all areas including the Mandir, Haveli and the Exhibition:
  ♦ Conduct: Please do not touch any of the carvings within the Mandir, Exhibition or Haveli. To Maintain the spiritual atmosphere observe silence inside the Mandir and the Exhibition.
  ♦ Dress Code: Casual Attire. No sleeveless tops. No shorts. No skirts shorter than knee length. Sarong will be provided upon request at the reception desk.

❖ 7:00 PM – Depart BAPS Hindu Temple and back to Crowne Plaza Toronto Airport Hotel

THURSDAY, APRIL 13, 2017

9:00 AM – 5:00 PM – BUS RIDE – TOUR & VISIT TO WORLD FAMOUS NIAGARA FALLS

• 9:00 AM – Bus Leaves Crowne Plaza Airport Hotel (80-Mile) to Niagara Falls. After arriving at the Falls, participants have three (3) hours to explore the following points of interest (on your own):
  ▪ Canadian Horseshoe Falls, The American Falls, Bridal Veil Falls
  ▪ Journey Behind the Falls
  ▪ Hornblower Niagara Cruises
Abstracts Of The Thirty-Ninth International Conference On Cement Microscopy

- White Water Walkway
- Whirlpool Aero Car
- Butterfly Conservatory
- Niagara’s Fury
- Queen Victoria Park (Adjacent to the Canadian Falls)
- Historical Museum at Lundy’s Lane
- The Maid of the Mist
- Marineland with Friendship Cove
- Skylon tower & Pavilion
- Canada One Factory Outlets
- Casino Niagara

Depending upon the availability of time, the Bus Tour and Visit to NIAGARA FALLS, might include the following Niagara Falls Nature & Gardens:

- Botanical Gardens
- Floral Clock
- Queenston Heights
- Oakes Garden Theaters
- Note: Click website: [www.niagaraparks.com/niagara-falls-attraction/](http://www.niagaraparks.com/niagara-falls-attraction/) for more information about the above-mentioned Niagara Falls Nature & Gardens

FACTS ABOUT NIAGARA FALLS

Niagara Falls, city in the Regional Municipality of Niagara, southeastern Ontario, Canada, a port on the Niagara opposite Niagara Falls, New York. The city overlooks the Horseshoe, or Canadian, Falls cataract of Niagara Falls; the crescent-shaped cataract is 54 M (177 ft.) high and carries nine times more water than its United States counterpart. Niagara Falls is an enormously popular tourist attraction, and it also serves as a major source of electricity for Ontario.

Points of interest include Queen Victoria Park, adjacent to the Canadian Falls and principal site of the annual Winter Festival of lights; the historical museum at Lundy’s Lane, site of the brutal battle between American and British forces in 1814; The Maid of the Mist, Marineland, with Friendship Cove which provides interaction with Killer and Beluga whales, an aquatic theatre and a game farm; Skylon Towe and Pavilion, containing a revolving restaurant and an observation deck overlooking the falls; the Butterfly Conservatory at the Niagara Parks Botanical Gardens; Canada One Factory Outlets which features brand name shopping; and of course, Casino Niagara. Niagara Falls covers a land area of 212 sq. km. (81.9 sq. mi.).
BRIEF FACTS ABOUT NIAGARA FALLS

- The word “Niagara” was derived from the Iroquois Indian word “Onguiaahra” meaning “The Strait.”
- Age of Niagara Falls: 12,000 Years
- Niagara Falls are the second largest falls in the world next to Victoria Falls in Southern Africa. The highest falls in the world are “Angel Falls” in Venezuela. What makes Niagara Falls so spectacular is the combination of their height and volume.
- Three different Falls make up what we call “Niagara Falls.” The Canadian Horseshoe Falls, The American Falls, and the Bridal Veil Falls.
- Number of tourists who visit the Falls every year: 12 million.
- Size of Falls: The rim of the Canadian Horseshoe Falls measures about 792 m (2200 ft.). The American Falls measure about 323 m (850 ft.). The Bridal Veil Falls measure about 15 m (50 ft.). Both the Canadian Horseshoe Falls and The American Falls measure 57 m (188 ft.) in height to the Niagara River below.
- Volume of water going over the American Falls: 567,810 L/second (150,000 gallons/second).
- Volume of water going over the Horseshoe Falls: 2,271,240 L/second (600,000 gallons/second).
- Source of water going over Falls: The 683,70 km² (264,000 square miles) of water that plunges over the Falls comes from the four of the great lakes: Superior, Michigan, Huron and Erie. Once the water flows over the Falls, it travels down the Niagara River to Lake Ontario, where it then flows into the St. Lawrence River and finally into the Atlantic Ocean.
- Deepest section of Niagara River: 52 M. (170 ft.) just below the Falls.
- Number of Nights The Falls are lit up: 365, During the Summer, the Falls are lit up between 9PM and midnight. At other times of the year, the Falls are lit up when it gets dark.
- Length of Whirlpool Rapids: 1.6 km. (1 mile). This is where the water drops 15m. (50 ft.), and speeds can reach as high as 9 meters per second (30 feet per second).
GUEST/SPouse PROGRAM

Guest/Spouse Program will be organized on demand for the 39th ICMA Conference in Toronto, Ontario, Canada and details will be shared as registration progresses and will be announced in the ICMA webpage (www.cemmicro.org). Please in your registration state if a spouse or guests will accompany you at the conference. (Spouse & Guest Committee: Lourdes M. Nisperos-Chairman; Maria Poellmann & JoAnn Buchanan-Co-Chairmen).

SEE YOU ALL AT THE 2018 ICMA CONFERENCE
VISIT THE ICMA WEBSITE FOR MORE INFORMATION ABOUT THE 40TH ICMA CONFERENCE
QUANTIFICATION OF SLAG DISSOLUTION IN SUPERSULFATED CEMENT

Rana Masoudi¹, R. Doug Hooton²

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ABSTRACT

Supersulfated cement (SSC) utilizes 80 – 85% slag, and around 10 - 12% calcium sulfate while using a minimal amount of Portland cement as an alkaline activator. The hydration of slag is very slow at early ages which results in slow rate of early strength development of SSC. In order, to improve the rate of early strength gain of SSC the rate of dissolution and hydration of slag in SSC need to be better understood. X-ray diffraction, TGA, and isothermal calorimetry is being used to quantify the hydration products of SSC. The amount of reacted slag was also estimated using an EDTA dissolution technique. However, this technique was found to be problematic and underestimated the quantity of slag reacted. To directly determine the amount of unhydrated slag in SSC, polished and carbonated paste samples at 1, 3, 7, and 28 days are being examined using backscattered electron imaging (BSE). The greyscale thresholding is being used to identify the amount of anhydrous slag. The volume fraction of unhydrated slag is being determined from the BSE images and then divided by the known volume fraction of slag in the SSC mixture to determine the percentage of slag reacted as a function of time. The results will be compared to that of the EDTA dissolution method to determine the most suitable technique for quantification of slag dissolution in SSC.
QUANTITATIVE ANALYSIS ON THE AMORPHOUS HYDRATED MAGNESIUM CARBONATE IN A CARBONATED REACTIVE MgO–PORTLAND CEMENT SYSTEM BASED ON ENERGY-DISPERSIVE X-RAY SPECTROSCOPY

Runxiao Zhang¹, Daman K. Panesar²

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ABSTRACT

Amorphous Mg(OH)₂ forms when MgO hydrates with the coexistence of calcium silicate hydrate, and its carbonation products in a reactive MgO-Portland cement (r-MgO-PC) system is likely to be an amorphous hydrated Mg carbonate with an estimated chemical formula of Mg(CO₃)₀.₈₆(OH)₀.₂₈•₀.₅H₂O. In this study, r-MgO-PC mortar specimens with r-MgO replacement of 20%, 40%, 60%, 80% and 100% were cast and carbonated for 28 days in an accelerated carbonation environment (95% relative humidity and 99% CO₂ concentration). Measurements of X-ray diffraction and thermogravimetry/differential thermal analysis were conducted to prove the existence of the amorphous hydrated Mg carbonate in the specimen with 60% r-MgO replacement. Measurements of energy-dispersive X-ray were conducted on a thin section made from the carbonated r-MgO-PC mortar with 60% r-MgO replacement. Eighteen measurements on nesquehonite (MgCO₃•3H₂O) were conducted, and the corresponding results were used to select appropriate references for the quantitative chemical composition analysis. Thirty-two measurements on the potential area of amorphous hydrated Mg carbonate and the results were compared with stoichiometric mass percentages of Mg in brucite (Mg(OH)₂), dolomite (CaMg(CO₃)₂), nesquehonite (MgCO₃•3H₂O), lansfordite (MgCO₃•5H₂O), hydromagnesite (Mg₅(CO₃)₄(OH)₂•4H₂O), dyepingite (Mg₅(CO₃)₄(OH)₂•5H₂O) and Mg(CO₃)₀.₈₆(OH)₀.₂₈•₀.₅H₂O. The comparative analysis reveals that the estimation of Mg(CO₃)₀.₈₆(OH)₀.₂₈•₀.₅H₂O is the closest Mg mass percentage to the real test result among all potential Mg carbonates. Moreover, with the quantity of hydrated water in Mg(CO₃)₀.₈₆(OH)₀.₂₈•₀.₅H₂O decreases, its Mg mass percentage is getting closer to the real test results, which leads to a potential extrapolate on the chemical formula of the amorphous hydrated Mg carbonate to be Mg₅(CO₃)₄(OH)₂•H₂O.

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POTENTIAL VALUE OF TRACE ELEMENTS IN THE SOURCING OF CEMENTITIOUS MATERIALS

Adrienne Weishaar¹, Remya P. N. Narayanan², Marion H. Emmert³, Aaron R. Sakulich⁴

¹, ⁴ Department of Civil and Environmental Engineering, Worcester Polytechnic Institute
², ³ Department of Chemistry and Biochemistry, Worcester Polytechnic Institute

ABSTRACT

Modern concrete production methods result in cementing phases with tightly controlled bulk chemistries, however, aggregates are selected mainly on the basis of regional availability. Since a wide variety of different minerals are used as aggregate, petrographic microscopy is the primary method used during the examination of concrete trace evidence in forensic contexts, whether in criminal cases, where concrete seems to be a material of choice for concealing evidence, or in civil cases regarding structural failures or disputes over workmanship. Petrographic microscopy is semi-quantitative and generally limited to determining whether the mineralogical classifications of aggregates are consistent between a sample and a potential source. Such analyses depend heavily on the experience of the investigator, and generally require relatively large samples and time- and resource-intensive sample preparation processes.

Although petrographic microscopy is a reliable tool for excluding evidence if the aggregate classifications are dissimilar, new, operator-independent, qualitative methods are needed for establishing positive relationships. The cementing phase, rather than the aggregates, may be of use to the forensic investigator. A wide variety of trace elements (those measured in the ppm range) can enter ordinary portland cement through a wide variety of paths: As impurities in raw or supplementary materials; through variations in plant processes or equipment; through the reuse of materials such as cement kiln dust; and through the use of alternative fuels. While the exact content of trace elements in the cementing phase of a concrete system can be changed based on mix design, it is hypothesized that the ratios of these elements one to another will serve as a unique ‘fingerprint’ that can be used to identify cementitious trace evidence. Samples of several ordinary portland cements, secured from geographically diverse facilities, were prepared through acid dissolution and investigated using Total X-Ray Fluorescence (TXRF). 23 trace elements were quantified in each sample, however, analyses indicate that only eight of these elements show the promise of being usable as a characteristic ‘fingerprint’. Two graphical interpretation methods (bar charts and ratio scatter plots), each display unique merits and flaws.
This initial report provides encouraging data, however, significant future research is necessary to establish the usefulness of trace element ratios to forensic investigators.
EVALUATION OF THE EFFECT OF OCEAN WATER ON THE HYDRATION OF COMMERCIAL MORTARS

Ruslan Liferovich, Mahir Dham, Kelly Hafkey, David Sobransingh

Mapei Corporation, Research and Development, Deerfield Beach, FL

ABSTRACT

In some marine environments where commercial mortars are used, there is a shortage of clean fresh water for hydration of the mortar, and multiple customers have inquired about mixing mortars with ocean water. While the effect of ocean water on the physical properties of commercial mortars has been widely studied, the chemical and mineralogical reaction pathways are not nearly as comprehensively understood. Therefore, in this study we evaluate the utility of ocean water for the hydration of cement systems. Two compositionally different cement systems were selected for study, one based on Portland cement and a second system based on a ternary binding system containing calcium aluminate cement, Portland cement and calcium sulphate. In order to gain insight into the reaction mechanism and pathways, the hydration was followed by X-ray diffraction, calorimetry and the mechanical resistance was also monitored up to 28 days.
EFFECT OF CELLULOSE NANOCRYSTAL (CNC) PARTICLES AS AN ADDITIVE ON THE RHEOLOGICAL BEHAVIOR OF OIL WELL CEMENT PASTE

Mohammad Reza Dousti¹, Yaman Boluk², Vivek Bindiganavile³

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² Professor, University of Alberta, yaman@ualberta.ca
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ABSTRACT

During the past few decades, oil and natural gas consumption has increased significantly. The limited amount of hydrocarbon resources on earth has led to a stronger desire towards efficient drilling, well completion and extracting. Well cementing is one of the most crucial and important steps in any well completion to fill the annulus between the casing string and the well bore. However, since it takes place at the end of the drilling process, a satisfying and acceptable job is rarely done. Hence, a large and significant amount of time and energy is spent in order to do the required corrections or retrofitting the well in some cases. Oil well cement paste needs to be pumped during the cementing process, therefore, the rheological and flow behavior of the paste is of great importance. This study examines the use of innovative cellulose-based nanomaterials on the flow properties of the resulting cementitious system. The cementitious paste developed in this research is composed of water, class G oil well cement, bentonite and cellulose nanocrystals (CNC), in which bentonite is used as a cross contamination component. Initially, the influence of CNC on the flow and rheological behavior of CNC and bentonite suspensions was assessed. Furthermore, the rheological behavior of oil well cement pastes dosed with CNC was studied using a steady shear parallel-plate rheometer and the results were compared to the rheological behavior of a neat oil well cement paste with no CNC. The parameters assessed were the yield shear stress and the viscosity. Significant changes in yield shear stress and viscosity were observed due to the addition of the CNC particles. Based on the findings in this study, the addition of a very small dosage of CNC to the oil well cement paste results in a more viscous cement slurry with a higher yield stress, demonstrating a shear thinning behavior.
REVENTION OF ASR IN OIL & GAS WELLBORE CEMENTS COMPRISING SILICA-BASED MICROSPHERES

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ABSTRACT

The oil and gas industry depends on a wide variety of cement additives to change the properties of cement slurries to fit requirements for each cementing operation. For example, when cementing in low pressure formations, such as those encountered at shallow depths offshore, it becomes necessary to use additive(s) to lower the density of the cement slurry, in order not to fracture adjacent rock formations. Foamed cement slurries have shown major defect due to bubble collapse at high pressures. Another, often-used additive is silicate-based microspheres. The microspheres are superior to foamed cements as they are designed to withstand the mechanical stresses placed on them in wellbore cements, as experienced in subsurface conditions, but extensive research has shown durability issues due to chemical instability. Basically in the high alkalinity environment of the cement, microspheres begin exhibiting alkali-silica reactivity (ASR). Typically, ASR becomes problematic as it develops against aggregate in concrete or mortar, where formation of an expansive silica gel results in creation of fractures. In wellbore cements containing silica-based microspheres, the mechanism of ASR is not clear. Limited publications show that the reaction product develops along the edge of the microsphere till it eventually reacts with the entire microsphere.

Prevention of ASR generally involves the use silica based materials throughout the cement matrix to act as a sink for alkalinity and restrain ASR expansion. This is traditionally achieved through the use of fly-ash, micro-silica, or slag. Recently, lithium nitrate has been shown to act as both a sink for alkalinity in the cement and create a beneficial product from reactions with Si, acting as a double prevention for ASR gel formation.

This study aims to investigate the onset and potential risks of ASR developed because of the addition of silicate-based microspheres in wellbore cements as compared to cements that typically exhibit negative effects of ASR. Additionally, this study will look at ASR mitigation in
cement containing silicate-based microspheres through the use of micro-silica, fly ash, and lithium nitrate. This is achieved by first creating a series of solutions of portlandite, silicate-based microspheres, sodium hydroxide, and potassium hydroxide to see the development of ASR. Then the same mixtures with additives of fly-ash, micro-silica, and lithium nitrate independently to see the how effective each are at preventing ASR.

Furthermore, cement samples will be constructed with silicate-based microspheres to allow the development of ASR, as well as fly-ash, micro-silica, and lithium nitrate individually. These samples will be tested for mechanical properties, physical properties, and micro-structure through the use of a micro-indenteter, porosimetry Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

Using this information, conclusions will be made on the mechanism of ASR attack on glass micro-spheres in wellbore cements and effectiveness of each additive used to prevent ASR in wellbore cements comprising of silica-based microspheres.
SCANNING ELECTRON MICROSCOPY EXAMINATION OF ALKALI-SILICA REACTION PRODUCTS IN THE ACCELERATED MORTAR BAR TESTING (ASTM C 1260)

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ABSTRACT

The accelerated mortar bar test (AMBT), adopted as ASTM C1260 and CSA A23.2-25A, is a rapid technique commonly used for examining the potential of aggregates to alkali-silica reaction (ASR). Unfortunately, the results obtained from this test are inaccurate in many cases. Some aggregates that show innocuous behavior in the field, or by the more accurate but time-consuming concrete prism test (ASTM C1293 and CSA A23.2-14A), are often identified as deleteriously reactive. These are referred to as false positives. Moreover, some reactive aggregates are incorrectly classified as non-reactive. These are referred to as false negatives. Seventeen aggregates with known ASR field performance were tested using the AMBT and, at the end of testing, thin-sections were prepared from the mortar bars. The microstructure, chemical composition, and spatial distribution of ASR gel were studied using scanning electron microscopy with energy dispersive x-ray spectroscopy. Signs of ASR were found in mortar bars that were associated with false positive results and very few or no signs of ASR in mortar bars that were associated with false negative results. In all specimens, the calcium content of the ASR gel was variable depending on its location; the calcium content increased as gel extended from the aggregate particles into the cement paste.

Keywords: Alkali-silica gel; Accelerated mortar bar test; ASTM C 1260;
PANEL DISCUSSION ON ASR: A 2017 PERSPECTIVE

AN ATTEMPT TO ESTIMATE PAST EXPANSION OF CONCRETE BASED ON PETROGRAPHIC STAGE OF ALKALI-SILICA REACTION

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ABSTRACT

This paper presents preliminary results of estimating the ratio of past expansion of ASR-affected concrete structures by two methods, and correlations with five petrographic stages of ASR: i) rim formation, ii) exudation of sol/gel around the aggregate, iii) cracking of aggregate, iv) extension of gel-filled cracks into cement paste, and v) map-cracking and precipitation of ASR gel along cracks into air voids distant from the aggregate. Past expansion was estimated by fitting the monitoring data with Larive’s equation defining the sigmoidal expansion curves (τl, τc, ε∞). Expansion of the intermediate stages iii) and iv) was interpolated from published data of concrete cylinder. Another method of estimating the past expansion was to measure the crack index, i.e. total crack width per unit length of concrete surface. By defining the past expansion and maximum crack width in terms of exponential functions, and by sub-dividing the advanced stage v) into several classes according to crack width, all the stages of ASR were arrayed on a log-linear scale of past expansion, irrespective of the rock type of the aggregate. Some Japanese volcanic gravel and sand produced past expansion >1% and ultimate expansion (ε∞) >3%, which were the largest without DEF ever reported. To improve precision, statistical treatments are necessary on the distribution and scatter of the crack width within and between structures in the future.

ASRによる過去のコンクリートの膨張量を岩石学的な進展段階をもとに推定する手法の一試案

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コンクリート構造物のASRによる過去の膨張率を二つの方法で推定し、岩石学的診断によるASRの進行段階との対応関係を予察した。ASRの岩石学的進行度は5段階で表示され、i) 骨材の反応リムの形成、ii) 骨材からのソル・ゲルの滲出、iii) 骨材内のひび割れの形成、iv) セメントベーストへのひび割れの進展、v) ベースト中の地図状ひび割れの進展、並びにひび割れに沿って骨材に近い気泡内のゲルの沈殿、よりなる。過去の膨張率は、1990年代に実施した岩石学的診断の直後に開始された長期モニタリングの膨張データの文献値を用いてLariveによる膨張のS字曲線（τl, τc, ε∞）をフィッティングし、建設時期まで遡って推定した。中間的な反応段階の膨張率は、コンクリートシリンダーの文献データで補完した。もうひとつの過去の膨張率は、構造物表面の単位長さ当りのひび割れ幅を計測し、得られたひび割れ指数より求めた。膨張率と最大ひび割れ幅をそれぞれ指数的に増加する数列として定義し、それぞれの区分を対数で等間隔になるように整理した。その結果、骨材の岩種にかかわらず、膨張が最も進展する進行度v)をひび割れ幅で細分することにより、ASRの全段階と過去の膨張率を対応づけることができた。日本の火山岩の砂利・砂の進行度v)では、岩石学的診断前の過去の膨張率は最大値1%以上で、建設後の終局膨張率の最大値が3%を超えたものがある。これはDEFのないASRの膨張率としては前例のない大きなである。推定精度の向上には、ひび割れ幅の分布状況やばらつきを考慮した統計的検討が必要であり、今後の課題としたい。
ABSTRACT

The current development in the global research on Alkali Aggregate Reactions (AAR) is presented. What is already known is highlighted, but still existing knowledge gaps are also looked upon. The focus is on how the reaction (or reactions) most likely is affected by the types of aggregates, level of humidity, amount of alkalis and the temperature. It is also discussed, how these parameters are affecting the outcome of the accelerated laboratory tests, both to classify reactivity of aggregates, but also to evaluate the performance of the various concrete mixes, with different types of binder etc.

Knowledge gaps include uncertainties if AAR is one type of reaction or a family of reactions; i.e. Alkali Silica Reaction and Alkali Carbonate Reaction. It is also still unclear how much of the quantity of available silica (quartz) participates in the reaction, and consequently the total magnitude and lifetime of the total reaction.

The complex inventory of alkalis in concrete is discussed in details. This includes the alkali leaching from concrete prisms in accelerated testing, along with the current ambiguous potential of alkali release from certain types of aggregates, and ways of determine that amount of alkali released. The verification of alkali release from aggregates, i.e. correlation of amount of alkali released in accelerated laboratory tests versus what is observed in real concrete, is still a challenge, and an issue of ongoing research.

The various test methods to determine AAR are presented, including challenges of quantification of reactive aggregates in the petrographic method, and the accelerated mortar bar test as a performance test, and testing time & critical limits in these types of tests. The challenge of alkali leaching in the concrete prism tests is discussed, along with assessment of the critical maximum temperature in such tests. These types of tests are also evaluated as performance tests, and
discussed how results from laboratory are comparable with results from concrete specimens from outdoor exposure sites.

Eventually, the activities and deliverables of the Technical Committees (TC) in RILEM (International union of laboratories and experts in construction materials, system and structures) are presented, along with presentation of the status of current working TC 258-AAA, which is focusing on the development of a performance based testing scheme for the prevention of deleterious AAR in futures structures.
PANEL DISCUSSION ON ASR: A 2017 PERSPECTIVE

PREVENTING ASR: WHAT ARE WE MISSING?

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ABSTRACT

The current Canadian guidance on ASR given in CSA A23.1/A23.2 was adopted in 2000. It led to development of similar guidance documents in the US, AASHTO R80 (formerly PP65) and ASTM C1778, as well as new developments in RILEM. These documents refer to test methods for determining potential deleterious reactivity and provide guidance on ASR mitigation using both performance and prescriptive approaches and are for the most part, more informative and useful than previous documents. Unfortunately, the ASTM C1260 rapid mortar bar test has a high rate of misdiagnosing an aggregate’s ASR potential but it is still widely used due to its rapidity. Until agencies and owners require aggregate producers to provide long-term but more reliable ASTM C1293 concrete prism expansion data, the use of some reactive aggregates will likely continue. As well, the ASTM C1567 and C1293 performance tests for demonstrating ASR mitigation do not allow the evaluation of the benefits of using low-alkali cements alone or in combination with SCMs. Current research at University of Toronto and elsewhere is attempting to address some of these shortcomings.
PANEL DISCUSSION ON ASR: A 2017 PERSPECTIVE

USE OF 2D/3D IMAGING AND OTHER CHARACTERIZATION TECHNIQUES FOR SERVICE LIFE PREDICTION AND MITIGATION OF ASR

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ABSTRACT

This presentation includes two sections. FIRST, preliminary data on the use of 2D SEM imaging/image analysis and 3D neutron and x-ray tomography is presented to quantify the degree of ASR reaction and damage that have occurred to date in an existing concrete structure. Measuring the degree of ASR with known accuracy is critical in predicting the remaining service-life and future performance of ASR-affected concrete. SECOND, SEM and other characterization techniques (i.e., thermogravimetric analysis, mercury porosimetry, and concrete pore fluid analysis) are used to evaluate the effectiveness and mechanisms of ASR mitigation using hydrated alumina. It is shown that hydrated alumina can successfully mitigate ASR as a result of (a) reducing silica dissolution at high pH through formation of a passivated alumino-silica layer, (b) reducing pH and alkalis in concrete pore solution, and (c) consuming and reducing dissolved Ca and portlandite in concrete.
SETTING CONTROL OF CALCIUM ALUMINATE CEMENT USING SUBSTITUTED ACETIC ACIDS AND THEIR CALCIUM SALTS

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ABSTRACT

Organic as well as inorganic additives are used to control the setting behaviour of calcium aluminate cements (CAC). Whereas LiCl and Li₂CO₃ typically act as accelerators, organic acids like citric, formic, acetic, chloroacetic, benzoic, naphthoic, glycolic, oxalic or tartaric acid and their alkaline earth salts retard the hydration reaction [1-13]. In the present study different derivatives of acetic acid (eq. 1) together with formic and acetic acid and the corresponding calcium salts were tested as admixtures to control the setting of Secar 51 as a typical calcium aluminate cement (CAC).

\[ H_{3-a}X_aCCOOH; \text{ with } a = 1, 2, 3; X = F, Cl, Br, I, \text{─CH}_3, \text{─CH}_2, \text{─OH, } =O \]  

All tested admixtures were found to act as retarders. Owing to the different effect of the substitutes on the acid strength and additional interaction potential with the cement paste they significantly affect the retarding effect. The delayed setting times of the onsets \( t_{\text{onset}} \) and of the heat low maxima \( t_{\text{max}} \) in calorimetry fit well by polynomial functions allowing a pre-adjustment of an intended setting time. By the exponential function \( \ln t_{\text{onset}} \) and \( \ln t_{\text{max}} = A \cdot e^{m} \) the retarding effect can be measured by \( m \) as a retardation power factor which only depends on the admixture type. This value \( m \) allows comparing the retarding effect of the different admixtures, e.g. in relation to their acid strength (Fig. 1). Among this, the \( \alpha \)-hydroxyl acids glycolic, glyoxylic, lactic and pyruvic acid were found to be the strongest retarders. For applied admixture concentrations \( c_{\text{admix}} \leq 0.05 \text{ M} \) the impact on the hydration products is minor. For higher concentrations the formation of the main hydration product \( \text{CAH}_{10} \) becomes reduced the stronger the setting is delayed and the Ca-richer phase \( \text{C}_2\text{AH}_{8-x} \) becomes increased accompanied with a small straëtingite formation, except for the \( \alpha \)-hydroxyl acids.

Crystallographic data of the calcium salts of the different tested admixtures are given to serve for phase identification of possible reaction products for higher admixture concentrations or for a possible use of the calcium salts as admixtures. A short overview of crystal structural features is presented giving hints on possible interactions of the substitutes with the cement paste (Fig. 1).
The strongest interactions by direct covalent bonds with calcium were found for the α-hydroxyl acetic acids also acting as the strongest retarders.

Fig. 1. Retardation power factor $m$ for onsets of setting of Secar 51 with admixtures in different molarities (left); crystal structure examples of calcium salts (right) demonstrate the different interactions with Ca$^{2+}$ and H$_2$O.

IN-SITU HYDRATION STUDY OF YE’ELIMITE-CONTAINING CEMENT BY PTYCHOGRAPHIC X-RAY COMPUTED TOMOGRAPHY

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ABSTRACT

Eco-cements are a desirable alternative to ordinary Portland cements because of their lower CO₂ footprints. For instance, the manufacture of Calcium SulfoAluminate (CSA) cements is more environmentally friendly than that of Portland cements as their production decreases CO₂ footprint by up to 40%. CSA cements contain ye’elimite, Ca₄Al₆O₁₂SO₄, as main phase. The hydration of ye’elimite leads to hydrated compounds: crystalline ettringite (AFt), crystalline monosulfoaluminate (AFm) and amorphous aluminum hydroxide gel, Al(OH)₃·nH₂O.

Here, we present the results of the study of the in-situ hydration of ye’elimite at different early ages by Ptychographic X-ray computed tomography (PXCT). PXCT is a nondestructive X-ray imaging technique which provides 3D mass density and attenuation coefficient distributions of cement pastes with an isotropic resolution close to 100 nm allowing distinguishing between mineralogical phases with very similar contrast. The sample was prepared by hydrating ye’elimite with gypsum in water (at a water-to-solid ratio of 1.4). Four datasets were recorded at 48, 53, 58 and 63 hours of hydration in the cSAXS beamline, at the Swiss Light Source. The main aim was to quantify the mass densities of the phases as well as the phase content evolution within this time interval. The different phases were identified and their mass densities determined. Furthermore, the tomograms have been segmented and the volume percentage of each phase have been determined and compared at the four different ages.

The study of the microstructures of the samples is very important and can be analyzed by PXCT. Different anhydrous particles were analyzed at all the selected ages to determine the reaction/dissolution rate of ye’elemite particles. Moreover, the evolution of the porosity with
time was also obtained in order to have a better understanding of the hydration mechanism of ye’elemite-containing cements.
STUDY OF THE EFFECT OF A NOVEL METAL SILICATE NANODISPERSION ON THE WATER PERMEABILITY OF CONCRETE

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ABSTRACT

The potential of a novel metal-silicate nanocomposite admixture to act as a permeability reducing agent is illustrated in this study. The admixture consists of mixed copper-calcium silicate hydrate nanoparticles that aggregate into fractal clusters characterized by a large specific surface area. Such clusters induce an autocatalytic C-S-H precipitation in the pore space. The different mode of C-S-H precipitation, in the presence of the admixture, can be observed both qualitatively, based on SEM imaging, and quantitatively based on radial distribution functions obtained from tomographic imaging.

Macroscopic observations based on water permeation measurements show that the permeability of concrete specimens is significantly reduced in the presence of the admixture. The fact that such a reduced permeability is also observed on cement pastes, in the absence of aggregates, suggests that permeability reduction is not related to any change in the characteristics of the interfacial transition zones, but rather on topological changes in the capillary pore network, resulting from the different mechanism of C-S-H precipitation. This hypothesis is confirmed by mercury intrusion porosimetry measurements, which show that although the total paste porosity does not significantly vary in the presence of the nanocomposite, a redistribution towards smaller pore sizes occurs.
CLUSTERING OF CEMENTITIOUS PARTICLES IN CONCRETE

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ABSTRACT

Cementitious paste homogeneity has been shown to be a significant factor in the quality of hardened concrete, potentially affecting strength, durability, appearance, and other physical properties. Occasionally, cementitious materials such as Portland cement, fly ash, slag, and microsilica can form clusters, either while still in the powdered state or once added to the concrete mix, within the cementitious paste matrix. These particle clusters are often quite dense, with very low water content, and do not appear to contribute to the formation of the resulting paste matrix as a whole- rather, they act as inert clusters of material. If a significant portion of the cementitious material remains bound up in these dense clusters, the resulting porosity (water to cementitious ratio) of the remaining of the paste will effectively be higher. This resultant higher porosity paste matrix can adversely affect the concrete in many ways, namely lowering the overall compressive strength; making the concrete more susceptible to freeze-thaw damage, physical attack, chemical attack, and chloride (for example from deicing salts) penetration; and/or resulting in an effective admixture overdose, which can affect set time, air contents, and placement. In addition it has also been reported deleterious alkali silica reaction (ASR) can develop from microsilica clusters behaving as reactive aggregate particles. This investigation focuses on three classic case studies that show different cementitious material clustering effects, which resulted in suboptimal concrete performance.
RAPID SCREENING METHOD TO FOR DETERMINING HYDRAULIC ACTIVITY INDEX OF PUMICEOUS POZZOLANIC MATERIALS FROM GUATEMALA, USING XRD ANALYSIS.

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ABSTRACT

One disadvantage of evaluating the quality of natural pumiceous pozzolans for cement production by traditional screening methods is the time lapse, of at least 28 days needed to calculate it. The main objective of this study was to determine if an XRD analysis could be used to have a faster and precise screening method of different pumiceous pozzolanic materials in the Guatemala region. A quantification of amorphous material (glass content) was measured using a Panalytical Empyrean XRD, using corundum as internal standard. We tested 37 samples from four different sources, 4 samples from the CCRL Pozzolan Program and 5 samples of natural quartz were included. Correlation between estimated amorphous content vs. ASTM C-311 hydraulic activity index showed an index of $r = 0.83$ & $r = 0.91$ for 7 and 28 days respectively. Those outcomes sustain the idea that XRD methods can be used to rapid screen the quality of pumiceous pozzolans used in cement production process. This study is relevant because it can be a faster way to filter data coming from different pozzolan sources, which can improve quality control decisions of the mining engineers and cement production processes and help manage them in an more efficient and effective way.
PARTICLE SIZE ANALYSES OF FLY ASH SOURCES BY LASER DIFFRACTION AND SECONDARY ELECTRON IMAGING

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ABSTRACT

Optimization of particle size distribution in coal fly ash is an important production parameter to improve fly ash quality and environmental aspects. The particle size distributions of fly ash from nine Canadian and Indian sources were measured using a Laser Diffraction Analyzer (LDA). Use of a LDA provided a meaningful characterization of particle size variability between the studied fly ashes. Scanning electron microscopy (SEM) was employed to visualize the distribution and shapes of the fly ash particles in a dry state. Each LDA measurement was supported by a corresponding scanning electron micrograph. SEM was a valuable tool for assessing and verifying the results of LDA measurements. Both LDA and SEM were found quick and efficient techniques in terms of sample preparation, data acquisition, and analysis. A bimodal size distribution was associated with all fly ashes as analyzed by LDA. The SEM micrographs showed two forms of particles: agglomerates and spherical primary particles.
ANALYSING HEAT FLOW AND SHRINKAGE OF A PORTLAND CEMENT MORTAR BY MULTIVARIATE STATISTICAL EXAMINATION OF XRD DATA

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ABSTRACT

In-situ XRD experiments are increasingly popular to investigate phase developments in reacting materials. In-situ XRD experiments create large amounts of data. For mortars it is a challenging task to extract all potentially interesting information from these in-situ XRD data by conventional methods (including the application of the Rietveld method). It is also often difficult to assess the significance of a feature in the data.

Multivariate statistics provides methods to examine large data sets. Exploratory factor analysis in particular can help to investigate data structures and to simplify the characterisation of these structures. It is therefore reasonable to apply multivariate statistics on the in-situ XRD data.

The hydration process of cement based materials becomes manifest in systematic changes in the in-situ XRD data. These systematic changes in the data (and therefore the hydration process) can be represented by a rather small set of (latent) variables. From a statistical point of view three such variables are sufficient to represent the non-random variations of the diffraction data of a hydrating Portland cement mortar.

It is possible to correlate the latent variables with material properties such as heat flow and shrinkage.

Heat flow and shrinkage curves can be considered as sum curves of several individual processes. The heat flow curve summarises the heat generation of several reaction such as the silicate reaction and the aluminate reaction. The shrinkage curve summarises dimensional change caused by several processes such as chemical shrinkage and drying shrinkage. It is shown that correlation analyses of diffraction data with heat flow or shrinkage data can be used to analyse time resolved the separate contributions of individual processes during the hydration.
USING OF BOTTOM ASH AND ALUMINIUM ANODIZING SLUDGE TO PRODUCE CALCIUM SULFOALUMINATE BELITE CEMENT

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ABSTRACT
A cement production generates high negative environmental impact, mainly associated to the CO₂ emissions. Calcium sulfoaluminate belite cement clinker (CSAB) has a lower content of calcium oxide, and the temperature required for its sinterization is ~200 °C lower than that used for Portland clinker). For these reasons, the CSAB is widely known as eco-friendly and low energy cement. However, the production process of CSAB-type cement requires a high amount of alumina which can be supplied mostly by bauxite. The higher demand of bauxite by other industries increase its cost and reduce its economical feasibility as a raw material. The Al₂O₃-rich wastes and their potential valorization and reuse might mitigate this economical and technical barrier for large-scale production. Alternative sources of alumina would add an economic and sustainable value to the final product. The objective of this study was to assesses the phases formation during the sinterization due the replacement of bauxite by bottom ash and aluminum anodizing sludge in CSAB clinkers produced in laboratory. Results showed that the mineralogy composition of CSAB clinker was strongly affected due to the addition of bottom ash. The amount of this waste replacing bauxite controls the formation of alite, belite, periclase, as well as the ye´elimite. Clinkers prepared from these replacement, are able to form up to 12.6% of alite (main phase Portland clinker), not normally found in CSAB clinkers, being sintered at 1250 °C. The results also elucidates the potential use of different residues as a Al₂O₃ source for the production of CSAB-type cement.
3D RECONSTRUCTION OF CRACK PLANE ORIENTATION FROM 2D SLICES OF CORES FROM REINFORCED CONCRETE PANELS AFTER SHEAR STRESS FAILURE

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ABSTRACT

Over the past decade, X-ray computed tomography (CT) methods have been widely used to characterize cracking in concrete. However, when using these methods the resolution of the images is a function of the size of the sample; the larger the sample, the poorer the resolution. Flourescent epoxy impregnation of 100 mm diameter cores, and subsequent slabbing, allows for the collection of detailed microscopic images that simultaneously capture both macro and micro level cracking; a feat not possible using traditional X-ray CT.
IMPACT OF TESTING METHODOLOGY ON THE DAMAGE MECHANISM OF SULFATE ATTACK IN PORTLAND CEMENT CONCRETE

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ABSTRACT

Upon penetration into concrete, sulfates can attack cement and its hydration products which can lead to formation of new calcium sulfate containing phases in the cement paste and consequently distress the system. Testing methodology plays a key role in the rate of ingress of sulfate ions and the formation of the new phases in the concrete. Physical changes, rate of ingress, and microstructural changes due to a continuous immersion method and an accelerated method were investigated. A modified version of ASTM C1012 was followed as the continuous method, while Swiss SIA 262/1 method which uses four cycles of drying and immersing was used as the accelerated testing methodology of sulfate attack evaluation in concrete. The acceleration method caused significantly higher rates of expansion, while propagation of cracks was more significant by the continuous immersion method.
MICROSCOPY OF 1860 NATURAL HYDRAULIC CEMENT IN CONCRETE FROM EAST BLOCK PARLIAMENT BUILDING

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ABSTRACT: The turn of the 19th century marks a transition in Canada’s cement manufacturing industry from traditional natural cements to the modern Portland cements that are ubiquitous today. In the late 1820s the construction of the Rideau Waterway prompted a search for local rock that could be burned to produce a water-tight cement, as opposed to importing English cement. Fortunately, a source was located in the township of Nepean (today part of Ottawa) where it was quarried and transported across the Ottawa River to kilns in nearby Hull (today part of Gatineau). Although the waterway was completed in 1832, the C.B. Wright & Co. kilns remained in operation up to at least 1893. As part of the on-going Parliament restoration, a sample of concrete from the East Block was examined microscopically and found to consist of a natural cement. Although many natural cement works were in operation at that time, it is likely that the closest available source (C.B. Wright & Co. Hull cement) was used. Natural cements were fired at temperatures much lower than Portland cement (1000 °C vs. 1400 °C) leaving tell-tale remnants of the original rock. These remnant natural cement grains are compared to a modern natural hydraulic cement produced by Rosendale Natural Cements.
A MICROSCOPY APPROACH TO FLY ASH BLENDED CEMENTS: QUALITY, DURABILITY AND PERFORMANCES

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ABSTRACT

Since the beginning of Portland cement manufacturing, the use of mineral additions that could reduce production costs has always been an important issue. Nowadays, considering the stringent requirements in greenhouses gases reduction, blended cements with low clinker content are mandatory and supplementary cementitious materials keep increasing their importance worldwide.

The present study is focused on fly ash addition, considering the quality, durability and strengths performances of highly substituted cements. The use of microscopy techniques, as well as powder diffraction analysis and mechanical testing, plays a key role in understanding how the hydration chemistry and the physical parameters control the macroscopic properties of this type of blended cements.
PETROGRAPHIC OBSERVATION AND LONG-DURABILITY OF CONCRETE
FROM SEA FORT NO. 2 CONSTRUCTED MORE THAN 100 YEARS AGO

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ABSTRACT
Sea Fort No2 is an artificial island constructed at the entrance of Tokyo Bay for the purpose of
defending the capital city of Tokyo. Construction started with mounding the foundation in 1889
(Meiji 22), installation of cannons in 1900 (Meiji 33) and was completed in 1914 (Taisho 3).
Present compressive strength of concretes taken from the north side concrete slope (constructed
1889 (Meiji 22)) on the foundation of the island, and the encircling wall of the battery cannon
(constructed 1907 (Meiji 40)), yielded about 30 N/mm². It was relatively high seeing that the
designed compressive strength at that time was 15 N/mm². Both concretes contained large
irregular voids and large cement particles (maximum size >200μm) rich in II type belite,
suggestive of a cement from a shaft kiln. Sand particles were fine-grained and round, occupying
a large part of concrete section. Unhydrated cement particles were coarsely grained, about the
same size as the fine aggregate. Hydration speed of those belite-rich cements was so slow that
the compressive strength developed gradually over 100 years. Neutralization depth of concrete
was also examined in this paper.

100年以上が経過した第二海堡に使用されたコンクリートの岩石学的観察および長期耐久性
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要 旨
第二海堡は戦時中、首都東京への敵艦隊の侵入を防ぐための要塞として東京湾に作られた人工島である。1889年(明治22年)
に人工島の基礎を築き、1900年(明治33年)に砲台などの上部構造物の拡張工事を開始し、1914年(大正3年)に完成した。砲台の基礎の砂の流出を防ぐための北側護岸の被覆コンクリート(1889年(明治22年)建立)と、カノン砲台
を囲む円形周壁のコンクリート(1907年(明治40年)建立)の現在の圧縮強度は30N/mm²であった。これは15N/mm²程度
の圧縮強度が想定されていた時代において、非常に高い値である。いずれの試料にも不規則な空隙が多く、セメントは水
和性の低II型ビーライトを多く含む緻密なものであり、粒子径は最大200μmと粗大で、細骨材の粒径とほぼ同じであっ
た。ビーライトに富む水和の遲いセメントが100年以上を経過し、ゆっくりと強度を発現してきたことが強度発現に
寄与したと考えられる。その他、中性化深さについても示す。
THE PICTON ONTARIO OUTDOOR EXPOSURE SITE – DAMAGE RATING INDEX ASSESSMENT AFTER EIGHTEEN YEARS OF ASR TESTING


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ABSTRACT

An alkali silica reactive coarse aggregate was used in a series of ternary blend silica fume and ground granulated blast furnace slag concrete outdoor slabs cast in the year 1998. After eighteen years of exposure, cores were retrieved and Damage Rating Indices performed on the slabs. In addition, alkali silica reaction products were characterized by optical microscopy and x-ray energy dispersive spectroscopy from the control (100% portland cement binder) concrete. The blended cement concrete successfully mitigated damage from expansive alkali silica reactivity as compared to the control.
UNIQUE POPOUTS CAUSED BY CALCINED DOLOMITE CONTAMINANT

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ABSTRACT

Occasionally, coarse aggregate used in various precast and cast-in-place concrete elements has been inadvertently contaminated with calcined dolomite pellets (CDP). Concrete with this contaminated aggregate can start exhibiting surface popouts within a few months after production due to the expansive hydration of the contaminant. A determination of the amount of this contaminant is important, as critical levels of CDP contamination can destroy a concrete element. Our studies included a methodology for the assessment of this type of contamination. This paper discusses historic work done to determine the levels of burned dolomite contamination and focuses on a recent project that included new studies. Cores extracted from affected concrete elements, samples of the contaminant, and concrete mix constituents were extensively studied using petrographic examinations and thermal analyses to assess the condition of the concrete and determine the content, distribution, and degree of hydration of offending CDP. The contaminant for the recent project was characterized as calcined (soft-burnt) dolomite, which is generally softer, more porous and more reactive than hard-burnt dolomite studied previously, and, consequently, is expected to complete its hydration and expansion in a shorter period. A supplementary analysis using standard-addition techniques was conducted to further refine the initial determination of the contaminant concentration. The amounts of CDP in the concrete elements were found to be relatively low, ranging from 0.03% to 0.25% by mass of concrete. A series of analytical studies allowed us to opine on the effect of the contamination on the functional performance and durability of the affected concrete elements. In the recent study, the concrete elements contaminated with CDP were not found to be structurally defective and should remain structurally durable over the intended design life, provided normal routine maintenance and timely repair of surface popouts are performed.

KEYWORDS: Popouts, Surface defects, Petrography, Microscopy, Thermal analysis, X-Ray diffraction, Calcined dolomite, Dolime, Dead burnt (hard burnt) dolomite, Calcined (soft burnt) dolomite, Free lime, Portlandite, Magnesium lime, Magnesia, Periclase, Brucite
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CDP-caused popout

Close-up view of the offending CDP

Cross sectional view of a CDP-caused popout

CDP particles

A CDP particle (plane-polarized light)

CDP particle (cross-polarized light)
MICROSCOPIC CHARACTERIZATION AS A MEANS TO INVESTIGATE THE MECHANISM OF BENEFICIAL CO₂ UTILIZATION

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ABSTRACT

A beneficial carbon dioxide utilization technology has been developed for ready mixed concrete production. Industrial installations have shown that carbon dioxide can be injected into concrete, while it is being batched and mixed, and a strength benefit, circa 15%, can arise. The performance is sensitive to the dose but the CO₂ is typically acting on the order of about 0.05% by weight of cement. A series of questions developed – how could such a small addition create a large performance change? Mechanistic understanding would require knowing what the reaction products look like, how they formed and what they were composed of.

Microscopic investigations have started to uncover the physiochemical process at work and build evidence for describing the mechanism. SEM was used to examine a model C₃S system and show that the action of the CO₂ is almost instantaneous – dissolution pits are visible on particle surfaces within 30 seconds of the dose. Reaction products consistent with amorphous calcium carbonate were examined though FIB sectioning and low energy EDS analysis of the chemical composition. A product examined using HRTEM showed what was found to be a mixture of amorphous and nano-crystalline phases. EELS was used in conjunction with high-angle annular dark-field STEM imaging to confirm the placement and presence of a Si rich layer around the formed carbonates. Atom Probe Tomography was used to examine the atomic composition of a reaction product in three dimensions.