Building Material Characterization by X-Ray Methods
Welcome

Today’s Topics
- Introduction
- Elemental analysis
- Phase analysis
- Rietveld in cement production control
- AXS CEM 2010 Symposium
- Selected papers from AXS CEM 2010
- Turn Key Cement Solution
- Summary
- Q&A

Arkady Buman
Business Development Manager

Alexander Seyfarth
Product Manager - XRF
Introduction

- Elemental analysis using XRF is now well established in the cement industry. Starting about 50 years ago, it took quite some time for XRF to displace traditional wet chemical methods.

- The benefit of XRF is the ability to analyze the pressed cement, raw mix sample directly, with high accuracy and excellent repeatability.

- Initially, XRF was purely a multichannel-based technique. Now, however, sequential techniques are also employed. Both are capable of performing cement analyses within 5 minutes.

- Instrument power and setup are defined by plant size and user preference for the sample preparation technique.

- With increased complexity of cement plants as well as number of kilns, laboratories are becoming more and more automated:
  - Combining automated sample preparation (mill and press) with one or more instruments
  - Automated sampling and pneumatic sample transport to the lab

- The drive for automation is anchored in the need for better quality and higher repeatability.
Elemental analysis of cement

- Performance requirements are defined by ASTM C114 (rapid test method) and/or ISO 29581 (EN 196-2)
  - See Bruker AXS Cement I and II on-demand webinars, where we detail the procedures and capabilities of WD-XRF with respect to these requirements: http://www.bruker-axs.com/webinars.html

- Classification of cement types is based on chemical and Bogue analysis results, as well as physical parameters and properties
  - See ASTM C150

- The chemical and physical properties are mainly governed by the phase composition, not by the elemental composition

- Approximation of phase composition by the Bogue equation is getting less and less accurate for “new” processes, such as the dry process and processes involving “non-traditional” fuels, raw materials or additives.
Why Bogue is not enough

- The Bogue calculation can only be a first estimation because it does not take into account solid solution effects and, additionally, there is no thermal equilibrium during clinker cooling.

- Neither the absolute values of Bogue can be right, nor relative changes can be monitored correctly, in particular if secondary fuels and secondary raw materials are used.

- Neubauer et al. (1997): Quantitative X-ray analysis of OPC Clinker by Rietveld Refinement, 10th ICCC
- Klaska et al. (2003): Effects of secondary fuels on clinker mineralogy, Cement International
How to obtain the real phase composition?

- **Microscopy**
  - manual point counting
  - direct method
  - accurate for main phases
  - time and labor intensive
  - not applicable for routine
  - requires experts

- **Powder XRD**
  - Usually Rietveld method
  - rapid
  - direct method
  - automated
  - accurate
Building Material Characterization by XRD

Arkady Buman
Rietveld analysis

X-ray diffraction with Rietveld analysis is currently the most powerful method for quantitative phase analysis.
EU is pioneering the use of XRD due to requirements for using secondary fuels and raw materials such as:

- Paints, solvents...
- Cows (disposal of mad cow carcasses)
- Recycled plastics

Combine XRD with the use of dry processes and pre-heaters, as well as targeted reduction of energy consumption

But it needs to be usable by non-PhD’s...
Please use your mouse to answer the question to the right of your screen:

For what are you using XRD already? (Select all that apply.)

- Freelime
- Qualitative analysis
- Phase identification
- Quantification of clinker
- Quantification of raw mix
- Quantification of cement
- I am currently not using XRD
History and basics of Rietveld analysis

Milestones

1966  7th Congress of the IUCr in Moscow
1977  Acceptance for X-ray diffraction
1983  Quantitative Phase Analysis
1999  TOPAS, the next generation of Rietveld software

Dr. Hugo M. Rietveld

Picture courtesy of Hugo Rietveld (http://home.wxs.nl/~rietv025/)
Poll Results

For what are you using XRD already? (Select all that apply.)

☐ Freelime
☐ Qualitative analysis
☐ Phase identification
☐ Quantification of clinker
☐ Quantification of raw mix
☐ Quantification of cement
☐ I am currently not using XRD

You said…
AXS CEM: A forum for information exchange and learning

Cement Symposia and Workshops

- 2000 Halle/Saale
- 2002 Karlsruhe
- 2003 Richmond/Virginia as part of the annual ICMA meeting
- 2004 Karlsruhe
- 2006 Wiesbaden
- 2010 Karlsruhe
Studies on cement hydration

Quantification of amorphous amounts
- slags, fly ashes and pozzulana are increasingly used as substitutes for clinker

Hotmeal phase analysis
- Control of the pre-heater efficiency
- Blockage prevention

Alite: distinction of modifications
- Chemical composition
- Reactivity
TOPAS
Total Pattern Analysis Solutions

- Next-generation Rietveld
- Fundamental Parameters Approach (FPA)
- No need for parameter turn-on sequence
- Large convergence radius
- Numerically stable
- Extremely fast
- Easy to use
- Fully automated version
  - Use fixed recipe
  - Press button, get result independent of operator
TOPAS
Accuracy in clinker analysis

- In phase analysis with XRD, we are talking about weight %, not about ppm as in XRF

- The accuracy of TOPAS clinker phase analysis has been proven and published in several publications

- In 2002 the National Institute of Standards recertified Portland Cement Clinker Reference Materials using TOPAS results

- The original NIST certification, which was released in 1989, was based mainly on microscopical point counting
Clinker

- Rietveld analysis provides true clinker composition
- Chemical and physical properties are mainly governed by the phase composition, not the elemental composition
- The Bogue calculation can only be a first estimation, because it does not take into account solid solution effects and, additionally, there is no thermal equilibrium during clinker cooling
- Neither the absolute values of Bogue can be right, nor can relative changes be monitored correctly, in particular if secondary fuels and secondary raw materials are used

- Neubauer et al. (1997): Quantitative X-ray analysis of OPC Clinker by Rietveld Refinement, 10th ICCC
- Klaska et al. (2003): Effects of secondary fuels on clinker mineralogy, Cement International
Rietveld analysis versus Classical parameters

The Deuna plant case study in 2003, done by POLYSIUS, Dyckerhoff and Bruker AXS, showed the benefits of Rietveld analysis for process optimization and control:

- trends and correlations can be seen, which are not provided by the classical parameters LSF (Lime Saturation Factor), Freelime and Bogue
- there is an existing upper limit, where an increase in LSF does not increase the real Alite (C3S) content
Benefits in quality and process control
Alite content versus LSF

(presented by Dyckerhoff Cement in 2004)
Benefits in quality and process control

Rietveld analysis versus Classical parameters

- There is no linear relationship between Alite content and LSF, as indicated by Bogue.
- Depending on the individual plant scenario, there is a maximum Alite level which cannot be exceeded by increasing the LSF.
- Generally, the clinker phase composition is more complex than indicated by Bogue.

An inappropriately high LSF results in wasting energy and raw materials!
Benefits in quality and process control

Clinker - degree of sulpharization

- High amounts of sodium and potassium lead to the formation of orthorhombic instead of cubic $C_3A$
- Both modifications have different reactivity and hydration behaviors
- If Na and K are fixed in alkali-sulfates, these elements are not available for the incorporation in $C_3A$
- By changing the degree of sulpharization, the ratio of $C_{3A_cubic}/C_{3A_{orthorhombic}}$ can be controlled
- XRD is the only method that can provide this information
Benefits in quality and process control

Clinker - degree of sulpharization

$C_3A$ in wt.%

$C_3A$ orthorhombic

$C_3A$ cubic

Degree of sulpharization
Benefits in Quality and Process Control

Clinker - degree of sulpharization

- The ratio $\text{C}_3\text{A}_{\text{cubic}}/\text{C}_3\text{A}_{\text{orthorhombic}}$ influences the water consumption of the cement

- Higher water consumption by $\text{C}_3\text{A}_{\text{orthorhombic}}$

- Direct effect on the cement performance
  - Porosity
  - Strength development
  - Durability
  - Etc.
Benefits in quality and process control

Summary - Clinker

- Effects can be seen with XRD which other methods do not provide (Bogue calculation, LSF, SR, AR)
- Control of the alkali mass flow (C$_3$A$_{cubic/orthorhombic}$, alkali-sulfates) is very important for cement setting behavior
- Operation closer to the limits of the process (saving of energy and expensive raw materials) instead of blindly following traditional parameters
- Freelime analysis can be done reliably with Rietveld
- Clinker grindability is related to Alite crystallite size
Please use your mouse to answer the question to the right of your screen:

What additives do you currently control in your plant or lab? (Select all that apply.)
- Limestone
- Slag
- Fly ash
- Puzzolan/volcanic ash
- Other
- None of the above
Benefits in quality and process control

Cement

- Control of the limestone addition (limestone filler)
  - Full spectrum approach vs. standard peak based calibrations

- Control of the cement mill operation
  - For example, dehydration of gypsum

- Control of blast-furnace slag addition in CEM II / CEM III
Poll Results

What additives do you currently control in your plant or lab? (Select all that apply.)
- Limestone
- Slag
- Fly ash
- Puzzolan/volcanic ash
- Other
- None of the above

You said…
MODERN CEMENT ANALYSIS

AXS CEM 2010

Dr. Marcus Paul

Dyckerhoff AG

Wilhelm Dyckerhoff Institut für Baustofftechnologie
ANALYTICAL METHODS APPLIED FOR CLINKER CHARACTERISATION

**Bogue**
- based on XRF
- rapid
- automated
- indirect
- inaccurate

C₃S = 4,073 x CaO - 7,602 x SiO₂ - 6,723 x Al₂O₃ - 1,422 x Fe₂O₃
C₂S = 2,867 x SiO₂ - 0,754 x C₃S
C₃A = 2,65 x Al₂O₃ - 1,691 x Fe₂O₃
C₄AF = 3,043 x Fe₂O₃

**Microscopy**
- manual point counting
- direct method
- accurate for main phases
- time and labor intensive
- not applicable for routine

**Rietveld method**
- based on XRD
- rapid
- direct method
- automated
- accurate

The Rietveld method offers additional structural information difficult to obtain by other analytical techniques.
RIETVELD METHOD AS A TOOL FOR QUALITY CONTROL BY PREDICTING MATERIAL PROPERTIES

**Composition**
- Quantitative phase assemblage
- Minor phases

**Crystallography**
- Polymorphism
- Solid solution
- Stress/Strain
- Crystallite size

**Clinker and cement characteristics**
- Strength development
- Setting time
- Reactivity
- Workability / Rheology

→ even quality as well as possibilities for optimization
POLYMORPHISM OF TRICALCIUM ALUMINATE ($\text{C}_3\text{A}$)

Low alkali content

- cubic
- less reactive
- slower hydration
- lower early strength

High alkali content

- ortho-rhombic
- more reactive modification (with potassium)
- quicker hydration
- higher early strength
POLYMORPHISM OF TRICALCIUM ALUMINATE (C$_3$A)

~ 10000 data points
POLYMORPHISM OF TRICALCIUM ALUMINATE (C₃A)

C₃A polymorphism affects cement properties:

• setting times
• early strength development
• workability

⇒ sulfate phases need to be adjusted
ALITE $M_1$-$M_3$: INFLUENCE ON STRENGTH DEVELOPMENT

The differences in the hydraulic properties of the $M_1$ and $M_3$ modifications were determined.

In the case of all the hydration periods monitored, the strength of cements with the $M_1$ modification was 10% higher than the strength of cements with the $M_3$ modification.

Cement and Concrete Research
Vol. 32, pp. 1169 - 1175
SUMMARY

• Rietveld allows a rapid determination of phase contents in clinker and cement
• Additional information about polymorphism of clinker phases
  ➞ Higher accuracy when all modifications are included in refinement
  ➞ Changes in polymorphism may occur slowly over long periods of time
  ➞ Polymorphism affects the hydraulic properties of cement
  ➞ Controlling polymorphism leads to even product quality
  ➞ Deliberately stabilizing a certain modification may improve properties
Benefits in quality and process control

Raw Materials, e.g. Natural Gypsum

<table>
<thead>
<tr>
<th>Material</th>
<th>Percentage</th>
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<tbody>
<tr>
<td>Anhydrite</td>
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<tr>
<td>Bassanite</td>
<td>3.89 %</td>
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<tr>
<td>Gypsum</td>
<td>49.71 %</td>
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<tr>
<td>Dolomite</td>
<td>12.47 %</td>
</tr>
<tr>
<td>Calcite</td>
<td>2.10 %</td>
</tr>
<tr>
<td>Magnesite</td>
<td>1.00 %</td>
</tr>
<tr>
<td>Quartz</td>
<td>1.01 %</td>
</tr>
</tbody>
</table>
Benefits in quality and process control

Raw Materials

- Quality control of gypsum
  - natural or artificial gypsum are often mixtures of gypsum, hemihydrate and anhydrite and other phases
  - Gypsum (CaSO$_4$·2H$_2$O), hemihydrate (CaSO$_4$·½H$_2$O) and anhydrite (CaSO$_4$) do have different reaction behavior
  - Easy distinction by XRD
- Quality control of limestone
- and any other crystalline sample
Step by step application of phase analysis for process optimization.

Urs Häseli
Holcim Switzerland, Plant Siggenthal
High concentrations of Ca-langbeinite lead to blockages in cyclone 1
Relationship of hotmeal phases and process problems.

High concentration of spurrite lead to blockages in kiln inlet.
**Analytical Trends in phase analysis**

**Studies on Cement hydration**

- Quantification of amorphous amounts
- Slags, fly-ashes and pozzulana are increasingly used to substitute clinker

**Hotmeal phase analysis**

- Control of the preheater efficiency
- Blockage prevention

**Alite: distinction of modifications**

- Chemical composition
- Reactivity

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**Relationship of hotmeal phases and process problems.**

**High concentration of sylvite lead to blockages in cyclone 2**
Observations from hotmeal phases

- Ca-langbeinite > 7%
- Spurrite > 7%
- Sylvite > 7%

High concentrations of different hotmeal phases create different buildups.
Reduction of cyclone blockages due to phase information

The kiln performance improved step by step.
Quantitative Rietveld analysis
Importance of sample preparation

- For quantitative X-ray diffraction, sample preparation has special requirements
- Overgrinding has to be avoided – phases like gypsum, hemihydrate, anhydrite and calcite are sensitive
- Sample preparation should be automated
- Two scenarios possible:
  - one procedure for XRD and XRF (possible with modern equipment)
  - Different procedures for XRD and XRF
TOPAS
Repeatability of analysis (values in wt.%)

<table>
<thead>
<tr>
<th></th>
<th>Alite</th>
<th>Belite</th>
<th>$C_3A_{ortho}$</th>
<th>$C_3A_{cubic}$</th>
<th>$C_3A_{sum}$</th>
<th>Ferrite</th>
<th>Periclase</th>
<th>Freelite</th>
<th>Arcanite</th>
<th>Quartz</th>
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<td>1.5</td>
<td>0.7</td>
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<td>4.6</td>
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</table>

The same sample was measured and analyzed ten times
(scan time: 4 min 37 sec; details in Lab Report XRD 59)
Same material, same control file... Different sample preparations
Rietveld Process Optimization

- Sampling and sample preparation optimization to:
  - Increase particle population and orientation randomization
  - Decrease preferred orientation
  - Avoid dehydration, destruction of some phase…

- Equipment parameters optimization to:
  - Good signal quality
  - Reasonable recording time

- Control file optimization to:
  - Adjust with local sample preparation process
  - Adjust with local materials
  - … assuming that starting control files is ok
Benefits in quality and process control

Cost Savings

- In almost every plant, a different scenario can be found.
- It is not easy to quantify savings in quality control:
  - How to quantify avoiding problems?
  - How to quantify that factors which were not apparent before, become visible now?
- We have seen in the last few years that the translation of XRD results into action is crucial.
- Therefore we don't provide numbers, but references.
- Return on investment typically is realized within two years.
Quantitative Rietveld analysis
Cement industry

“The question is not IF, but WHEN the Rietveld method will be implemented for quality optimisation and process control in ALL cement plants.”

Paul, M. (2004), Dyckerhoff Cement (Buzzi Unicem)
Summary

- It is now possible to optimize and control the cement-making process
- The tools which allow this to happen are:
  - XRD instrument designed for fast process control
  - TOPAS software (the next generation Rietveld!)
  - Repeatable sample preparation
  - Know-how

Only TOPAS produces results automatically, independent of the operator

Using XRD with TOPAS, the cement manufacturer will be able to produce better quality cement and realize cost savings in doing so.
The Bruker AXS Cement Solution

Alexander Seyfarth
Fast, optimized process control

- automatic sampling  ✔
- automatic preparation  ✔
- automatic measurement  ✔
- automatic phase quantification  ✔
The Bruker AXS Cement Solution

Instrumentation, Software and Know-How

- D4 ENDEAVOR with LYNXEYE detector – ideal for fully automated analysis and high sample throughput
- TOPAS: the next generation of Rietveld software
  - Fundamental Parameter Approach (FPA)
  - Large convergence radius
  - Extremely fast calculation (less than 5 seconds for clinker analysis)
  - Stable
- Application Know-How
  - Dedicated application experts (XRF, XRD) for turn-key operation
    - US: Arkady Buman
    - International: Rainer Schmidt
The Bruker AXS Cement Solution

D4 ENDEAVOR and LYNXEYE detector
more than a process diffractometer

- The fastest instrument available
- Automation-ready via robot or conveyor belt
- Large magazine allows intelligent and flexible sample handling
Bruker AXS Cement Solution
Integrated sample preparation for XRD and XRF

Transfer of results via LAN, e.g. to plant control system, LIMS system
The Bruker AXS Cement Solution

TOPAS Total Pattern Analysis Solution

Benefits:
- Reliable results
- Does not break down when sample composition changes
- Best available reproducibility

Next generation Rietveld software
Bruker AXS Cement Solution

- Simply click a button to start a sample or a batch job
- Configurable by the user
## Bruker AXS Cement Solution

### System Joblist

<table>
<thead>
<tr>
<th>Sample</th>
<th>Position</th>
<th>Method</th>
<th>Preparation</th>
<th>State</th>
<th>XRF State</th>
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### Result Table

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<td>3.36</td>
<td>1.68</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Average:**
- 5.26% Al2O3, 1.88% Fe2O3, 0.14% MgO, 1.88% SiO2, 0.36% CaO, 1.68% SO3, 3.36% AI2O5, 1.68% Fe2O5

**Standard deviation:**
- 0.01, 0.00, 0.01, 0.01, 0.01, 0.00, 0.00, 0.00

**Maximum:**
- 5.20% Al2O3, 1.88% Fe2O3, 0.11% MgO, 1.88% SiO2, 0.36% CaO, 1.68% SO3, 3.36% AI2O5, 1.68% Fe2O5

**Minimum:**
- 5.15% Al2O3, 1.87% Fe2O3, 0.11% MgO, 1.87% SiO2, 0.35% CaO, 1.64% SO3, 3.35% AI2O5, 1.64% Fe2O5

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**Operator:** Bandic
**System message:** -
Bruker AXS Cement Solution

- Push button analysis: from pickup to result in < 5 minutes! As fast as XRF! No user interaction!
- TOPAS ensures results even with changing materials, no reference material required!
- Quantitative XRD enhances the ability for process and quality control. Real phases equals to real information
  - Information is power
- Cost savings will cover instrument investment
  - Individual Payback calculation, ask the references!
- Get your turn-key solutions from the pioneering experts
Any Questions?

Please type any questions you may have in the Q&A panel and then click Send.
Visit us at:

20th Industrial Minerals International Congress & Exhibition, Mar 21-24, Miami, FL

International Cement Microscopy Association (ICMA), Mar 28 - Apr 1, New Orleans, LA

Cement Chemists’ Society (CCS) XRF/XRD Workshop, Jun 8-10, Dallas, TX

Denver X-ray Conference
Aug 2-6, Denver, CO

Cement Chemists’ Society (CCS) Annual Meeting, Oct 18 - 22, Orlando, FL

www.bruker-axs.com
For More Information

- Contact Arkady Buman and register for one of our upcoming seminars:

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Jun 8-10  Atlantic City, NJ

**TOPAS Seminar West**  
Jun 15-17  Las Vegas, NV

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Thank you for attending!

Please take a moment to complete the brief survey on your screen. Your feedback is very important to us.

Copies of this presentation and related AXS CEM 2010 materials will be emailed to you.