XRD and Cements: from research to control quality

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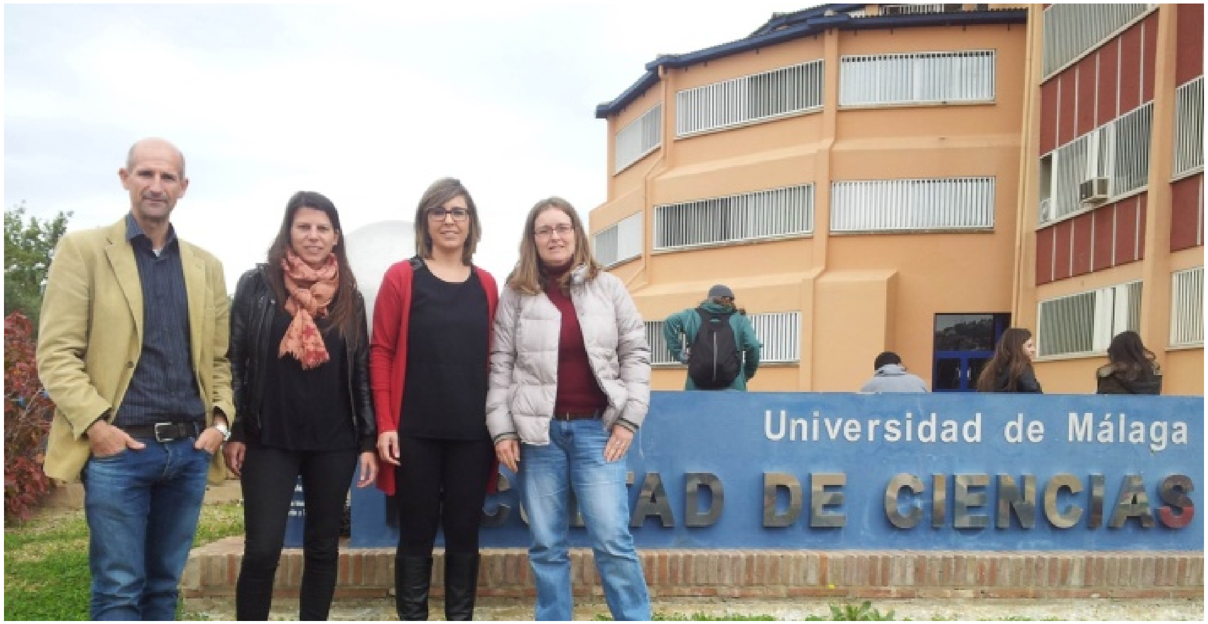
Spin off: X-Ray Data Services

X-RAY DATA SERVICES



Analysis

(Phase analysis, Amorphous determination)

Innovation & Knowledge transfer

(Research collaborations, Project assessment, ...)

X-Ray Data Services

Training

(Data collection, Analysis interpretation, ...)

Consulting

(Protocol validation, Regulations, ...)

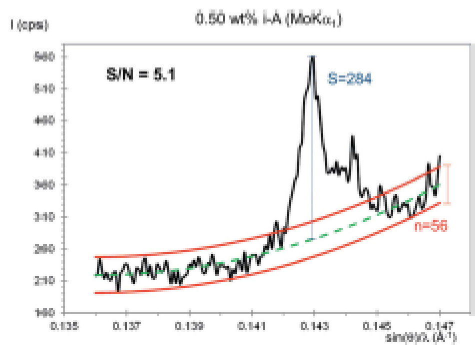
Research

research papers

JOURNAL OF APPLIED CRYSTALLOGRAPHY
ISSN 1600-5767

Accuracy in Rietveld quantitative phase analysis: a comparative study of strictly monochromatic Mo and Cu radiations

L. León-Reina,[a] M. García-Maté,[b,c] G. Álvarez-Pinazo,[b,c] I. Santacruz,[b] O. Vallcorba,[d] A. G. De la Torre[b] and M. A. G. Aranda[b,d]*



OPEN ACCESS

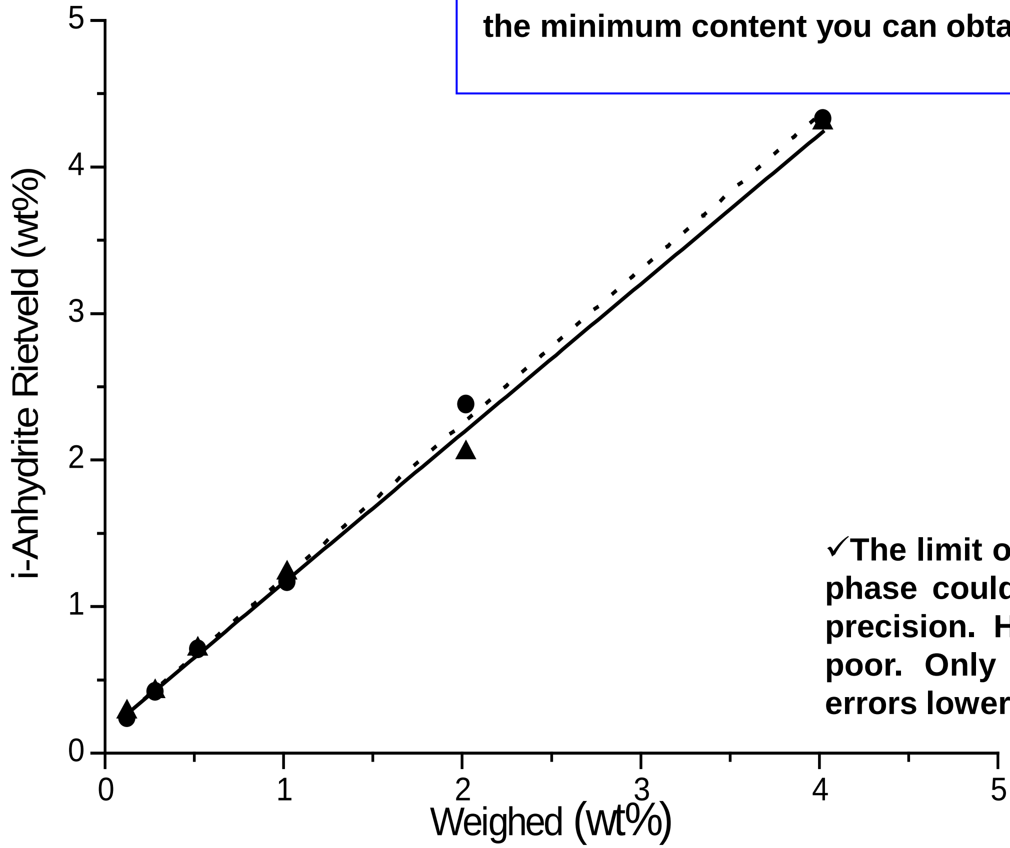
J. Appl. Cryst. (2016) 49, 722–735

Research

Crystalline inorganic mixtures:

A matrix of: [calcite (C) + gypsum (Gp) + quartz (Q)] +
(0.00, 0.12, 0.25, 0.50, 1.0, 2.0 and 4.0 wt%) insoluble Anhydrite (i-A).

Limit of Quantification LoQ
the minimum content you can obtain three times larger than its associated standard deviation.



	Mo-Kα_1 trm.	Cu-Kα_1 refl.
R^2	0.997	0.998
Slope	1.02(3)	1.05(2)
Intercept	0.14(5)	0.14(4)

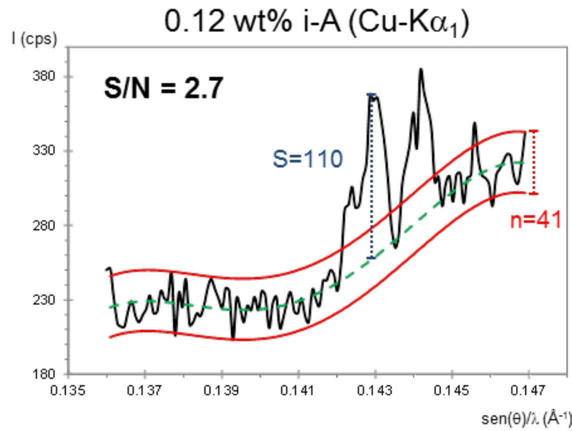
✓**The limit of quantification (LoQ) for a well crystallized inorganic phase could be established to be close to 0.12 wt% with good precision. However, the accuracy of these analyses was quite poor. Only contents >1.0 wt% yielded analyses with relative errors lower than 20%.**

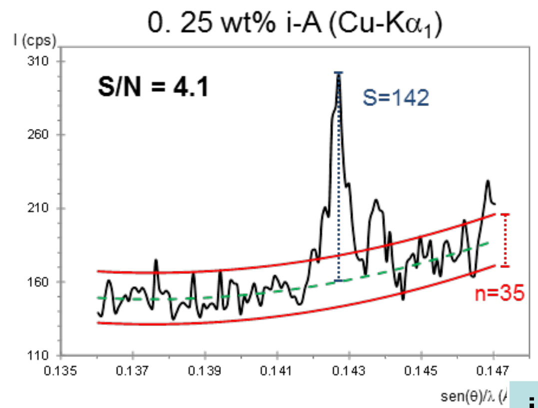
J. Appl. Cryst. (2016) 49, 722–735

Crystalline inorganic mixtures:

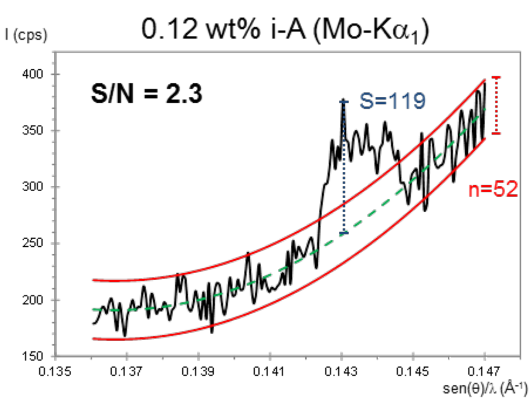
A matrix of: [calcite (C) + gypsum (Gp) + quartz (Q)] + (0.00, 0.12, 0.25, 0.50, 1.0, 2.0 and 4.0 wt%) insoluble Anhydrite (i-A).

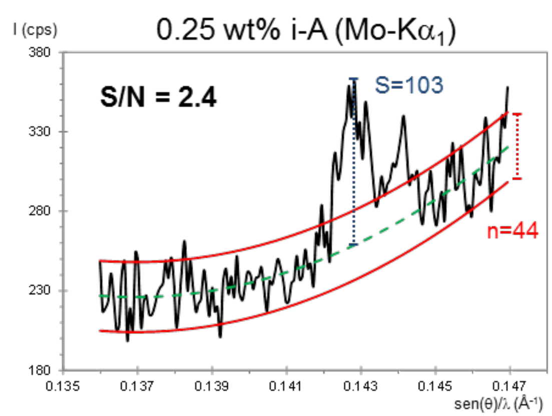
Limit of Detection LoD the minimum amount of the analyte yielding a powder pattern with its strongest (not overlapped) diffraction peak with an S/N larger than 3.0.

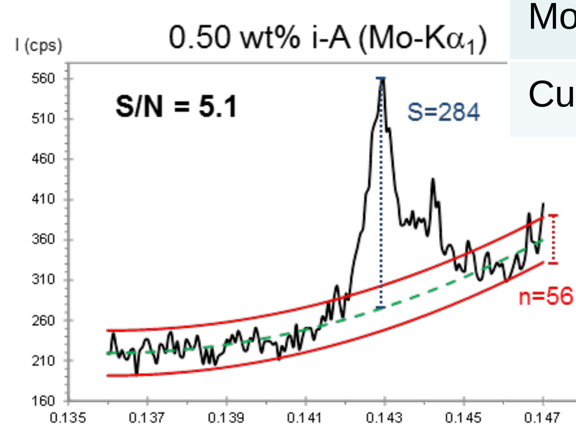




i-A	LoD
$MoK\alpha_1$	0.3 wt%
$CuK\alpha_1$	0.2 wt%







J. Appl. Cryst. (2016) 49, 722–735

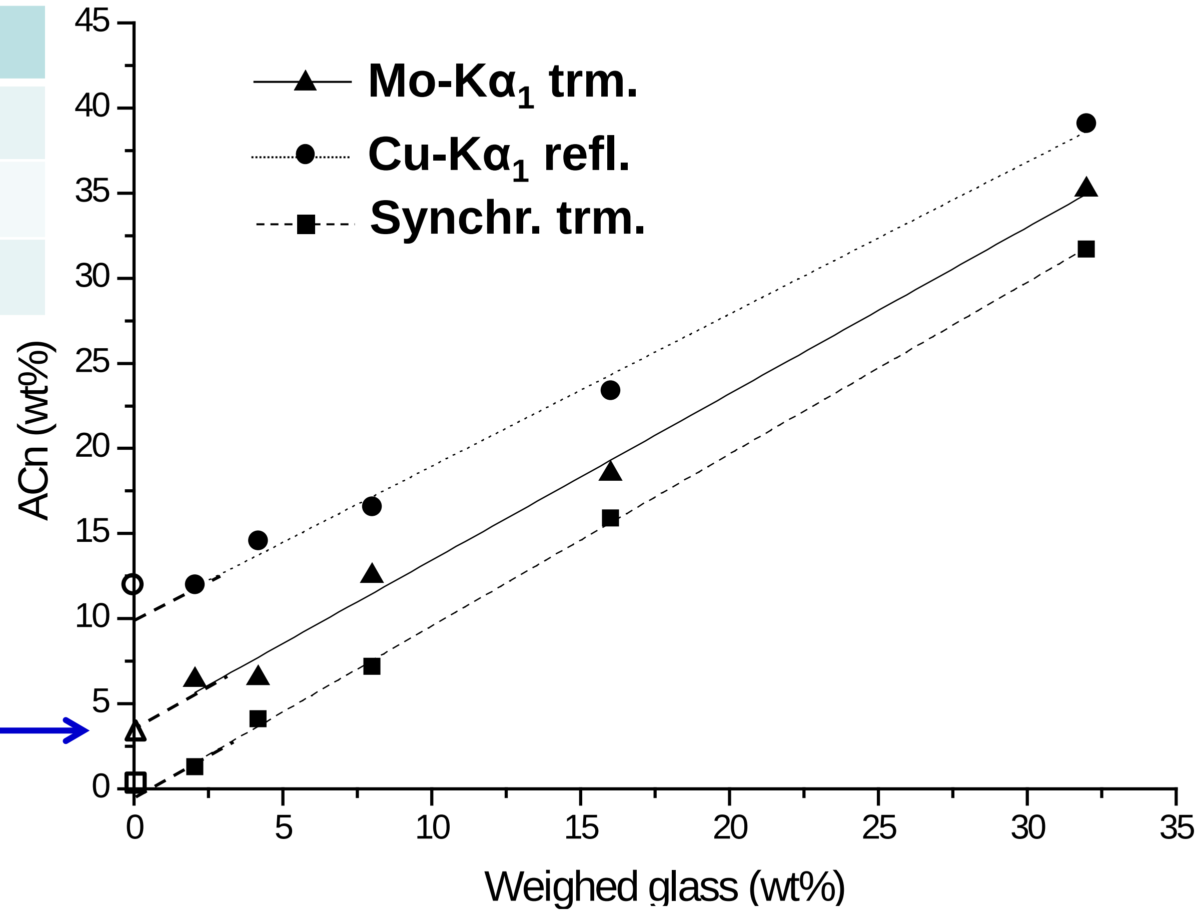
Variable amorphous contents within an inorganic crystalline phase matrix:

[calcite (C) + zincite (Z)] + (0, 2, 4, 8, 16 and 32 wt%) amorphous ground glass (Gl).
Internal standard (20 wt% Quartz (Q)).

Sample 0.0 wt% glass	ACn /wt%
Synchrotron	0.4(1)
MoKα_1	3.5(1)
CuKα_1	12.0(1)

MoKα_1 results were more accurate

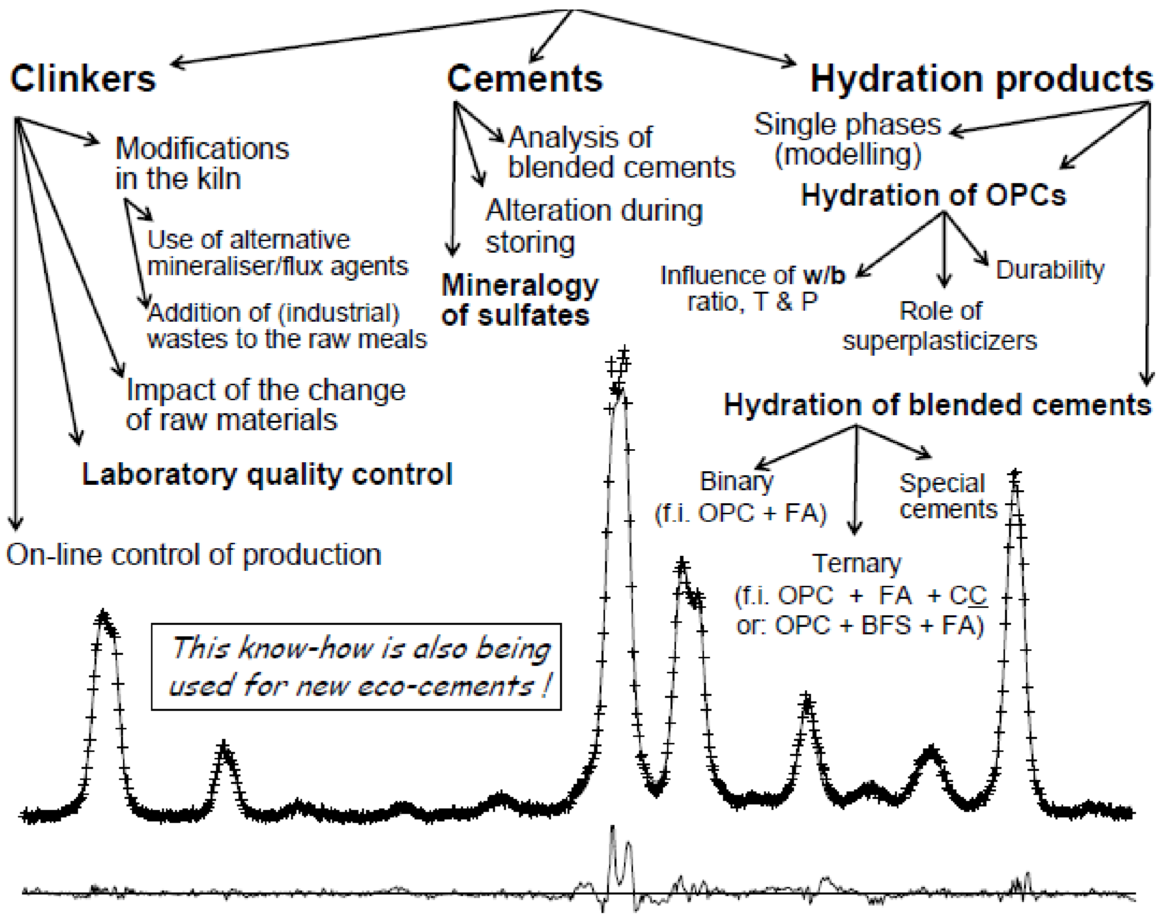
- ✓ **Slope closer to 1.0**
- ✓ **Small value in the amorphous free sample**
- ✓ **Good aggrement**



J. Appl. Cryst. (2016) 49, 722–735

RQPA gives useful information for cements.

(The answer will depend on the problem)



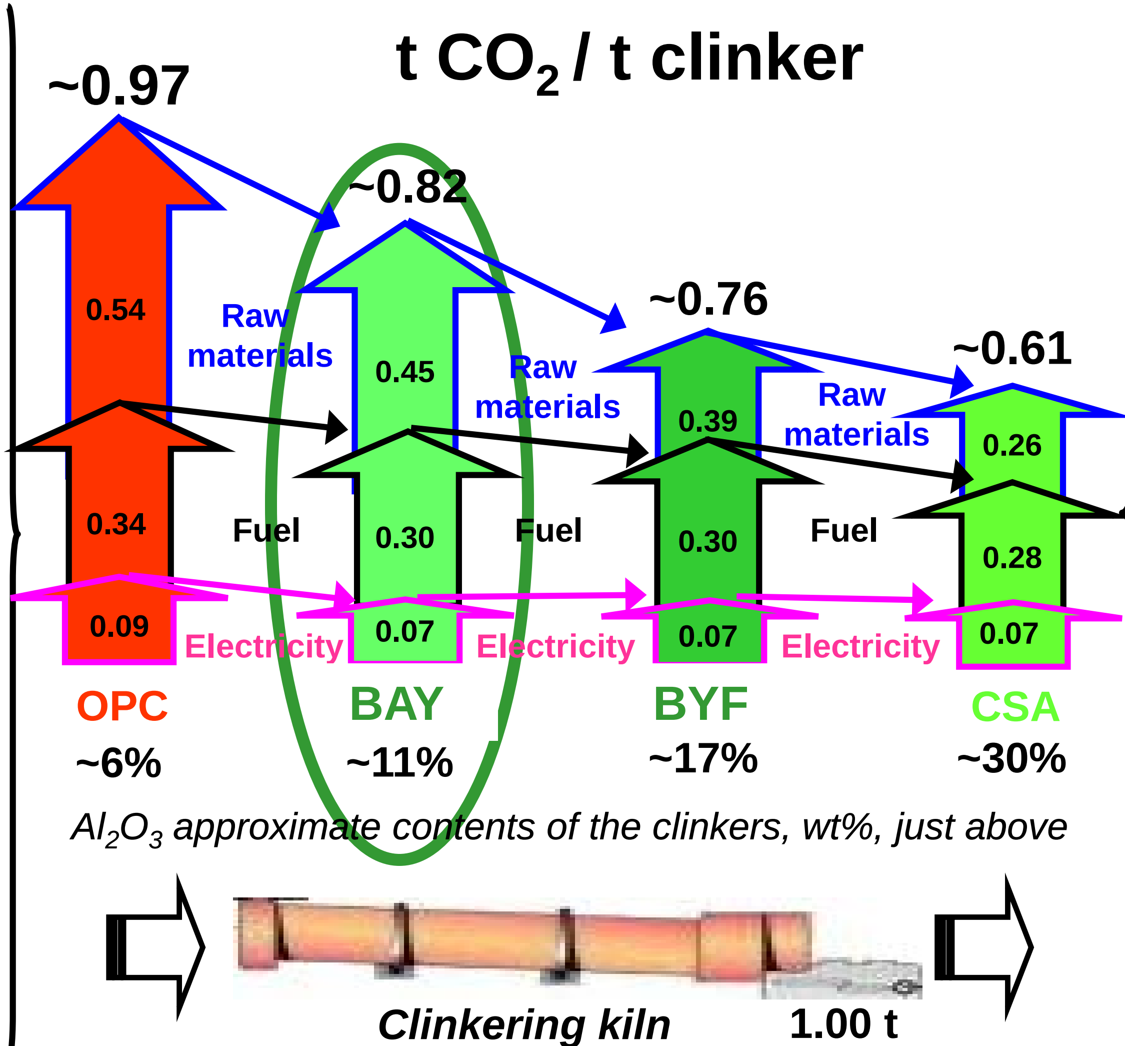
Cement research @ UMA > eco-cements

OPC
Raw materials
78 wt% calcite
10 wt% kaolin
10 wt% quartz
2 wt% iron oxide

BAY
Raw materials
69 wt% calcite
19 wt% kaolin
6 wt% quartz
3 wt% iron oxide
3 wt% gypsum

BYF
Raw materials
59 wt% calcite
9 wt% kaolin
7 wt% quartz
17 wt% red bauxite
8 wt% gypsum

CSA
Raw materials
35 wt% calcite
9 wt% kaolin
5 wt% red bauxite
26 wt% white bauxite
25 wt% gypsum



OPC clinker
Phases
65 wt% C_3S
15 wt% C_2S
10 wt% C_4AF
10 wt% C_3A

BAY clinker
Phases
55 wt% C_2S
15 wt% C_3S
15 wt% $C_4A_3\underline{S}$
15 wt% C_4AF

BYF clinker
Phases
50 wt% C_2S
30 wt% $C_4A_3\underline{S}$
20 wt% C_4AF

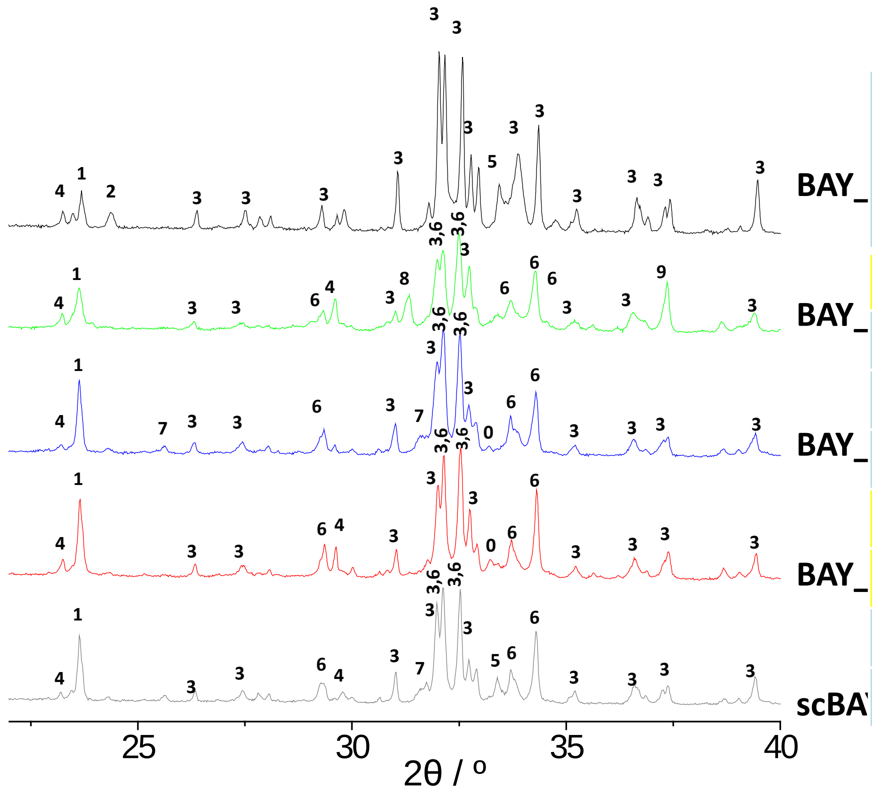
CSA clinker
Phases
65 wt% $C_4A_3\underline{S}$
20 wt% C_2S
6 wt% C_4AF
9 wt% $C\underline{S}$

Cement research @ UMA > eco-cements BAY

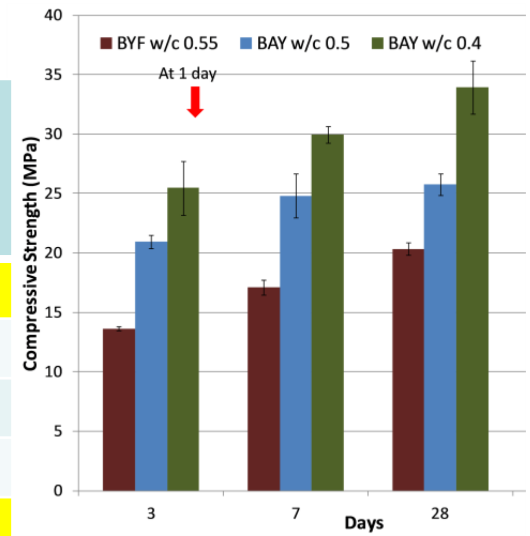
Manuscript submitted to *Cement and Concrete Composites*

Clinkering and hydration of Belite-Alite-Ye´elimite cement

Londono-Zuluaga, D.[1,2], Tobón, J.I.[2], Aranda, M.A.G.[1,3], Santacruz, I.[1], De la Torre, A.G*[,1]

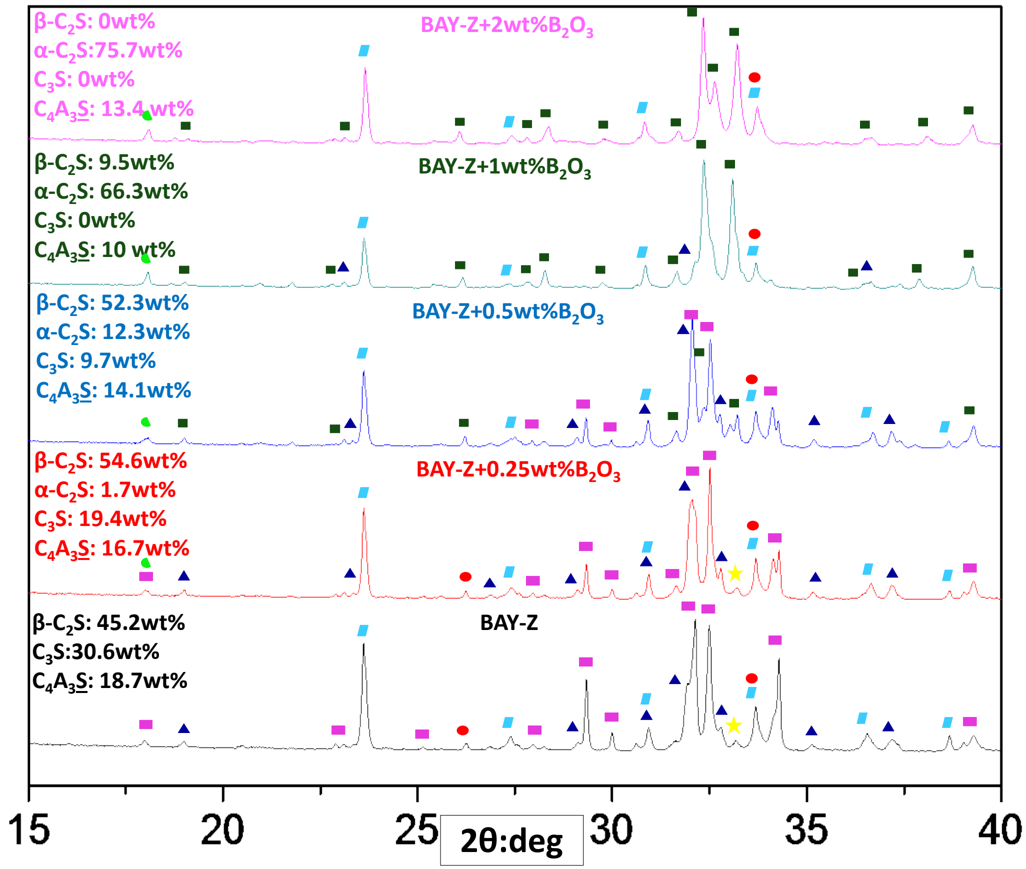


Phase	1300ºC/15min with excess of sulfur*
ß-C_2S	59.4(2)
γ-C_2S	1.2(1)
C_4AF	6.5(2)
$C\hat{S}$	0.5(1)
o-$C_4A_3\hat{S}$	10.4(1)
C_3S	14.3(2)
$C_{12}A_7$	4.5(1)
Fluorellestadite	2.6(1)



Cement research @ UMA > eco-cements BAY

Synthesis of activate Belite-Alite-Ye'elimite clinker (BAY)
Manuscript in progress



The effect of different quantities of B_2O_3 like dopant to obtain activate BAY clinkers with α'_H-belite and pseudo-cubic-ye'elimite, jointly with alite.

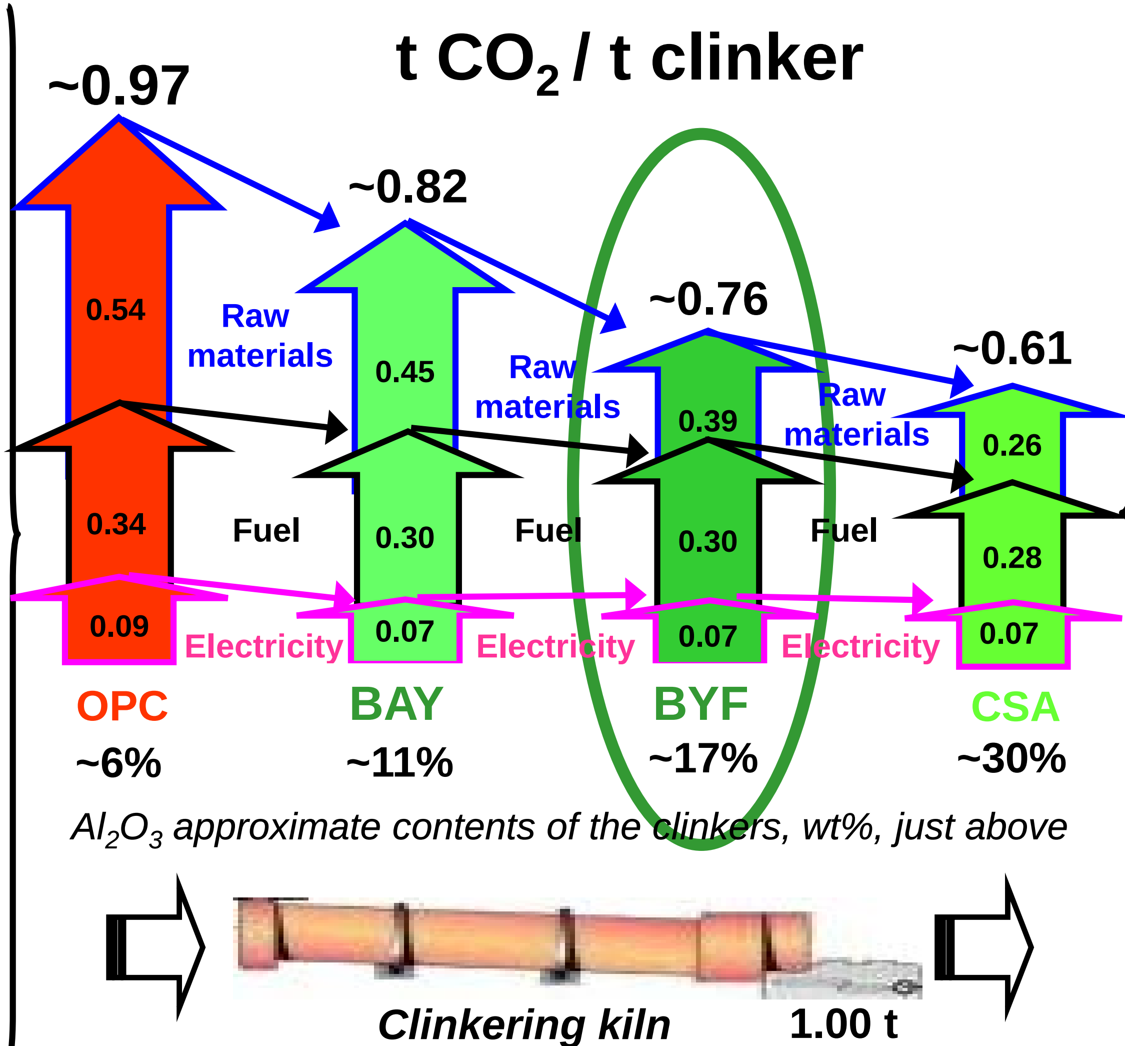
Cement research @ UMA > eco-cements

OPC
Raw materials
78 wt% calcite
10 wt% kaolin
10 wt% quartz
2 wt% iron oxide

BAY
Raw materials
69 wt% calcite
19 wt% kaolin
6 wt% quartz
3 wt% iron oxide
3 wt% gypsum

BYF
Raw materials
59 wt% calcite
9 wt% kaolin
7 wt% quartz
17 wt% red bauxite
8 wt% gypsum

CSA
Raw materials
35 wt% calcite
9 wt% kaolin
5 wt% red bauxite
26 wt% white bauxite
25 wt% gypsum



OPC clinker
Phases
65 wt% C_3S
15 wt% C_2S
10 wt% C_4AF
10 wt% C_3A

BAY clinker
Phases
55 wt% C_2S
15 wt% C_3S
15 wt% $C_4A_3\underline{S}$
15 wt% C_4AF

BYF clinker
Phases
50 wt% C_2S
30 wt% $C_4A_3\underline{S}$
20 wt% C_4AF

CSA clinker
Phases
65 wt% $C_4A_3\underline{S}$
20 wt% C_2S
6 wt% C_4AF
9 wt% $C\underline{S}$

Cement research @ UMA > eco-cements BYF

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X-RAY DATA SERVICES

Advances in Cement Research
Volume 28 Issue 8

Hydration of belite–ye'elimite–ferrite cements with different calcium sulfate sources
Álvarez-Pinazo, Santacruz, Aranda and De la Torre

Advances in Cement Research, 2016, **28**(8), 529–543
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Paper 1600030
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Published online ahead of print 18/07/2016
Keywords: compressive strength/gypsum (hemihydrate, anhydrite)/hydration

ice Institution of Civil Engineers publishing

Hydration of belite–ye'elimite–ferrite cements with different calcium sulfate sources

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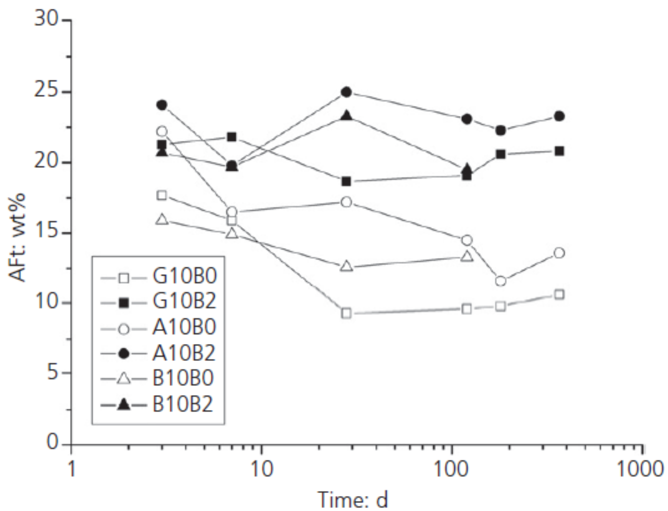

Figure 3. Amount of AFt determined by Rietveld method with time for all the studied cements

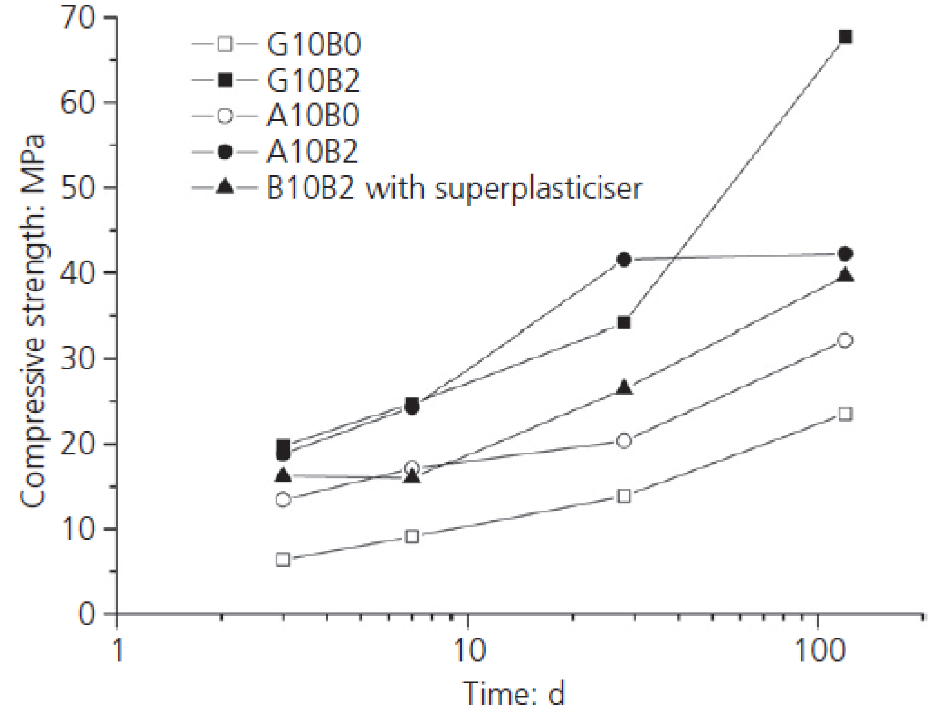

Figure 8. Compressive strengths of mortars prepared from all pastes at different hydration times (3, 7, 28 and 120 d)

RQPA & G-Factor

Cement research @ UMA > eco-cements

OPC
Raw materials
78 wt% calcite
10 wt% kaolin
10 wt% quartz
2 wt% iron oxide

BAY
Raw materials
69 wt% calcite
19 wt% kaolin
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3 wt% iron oxide
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BYF
Raw materials
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CSA
Raw materials
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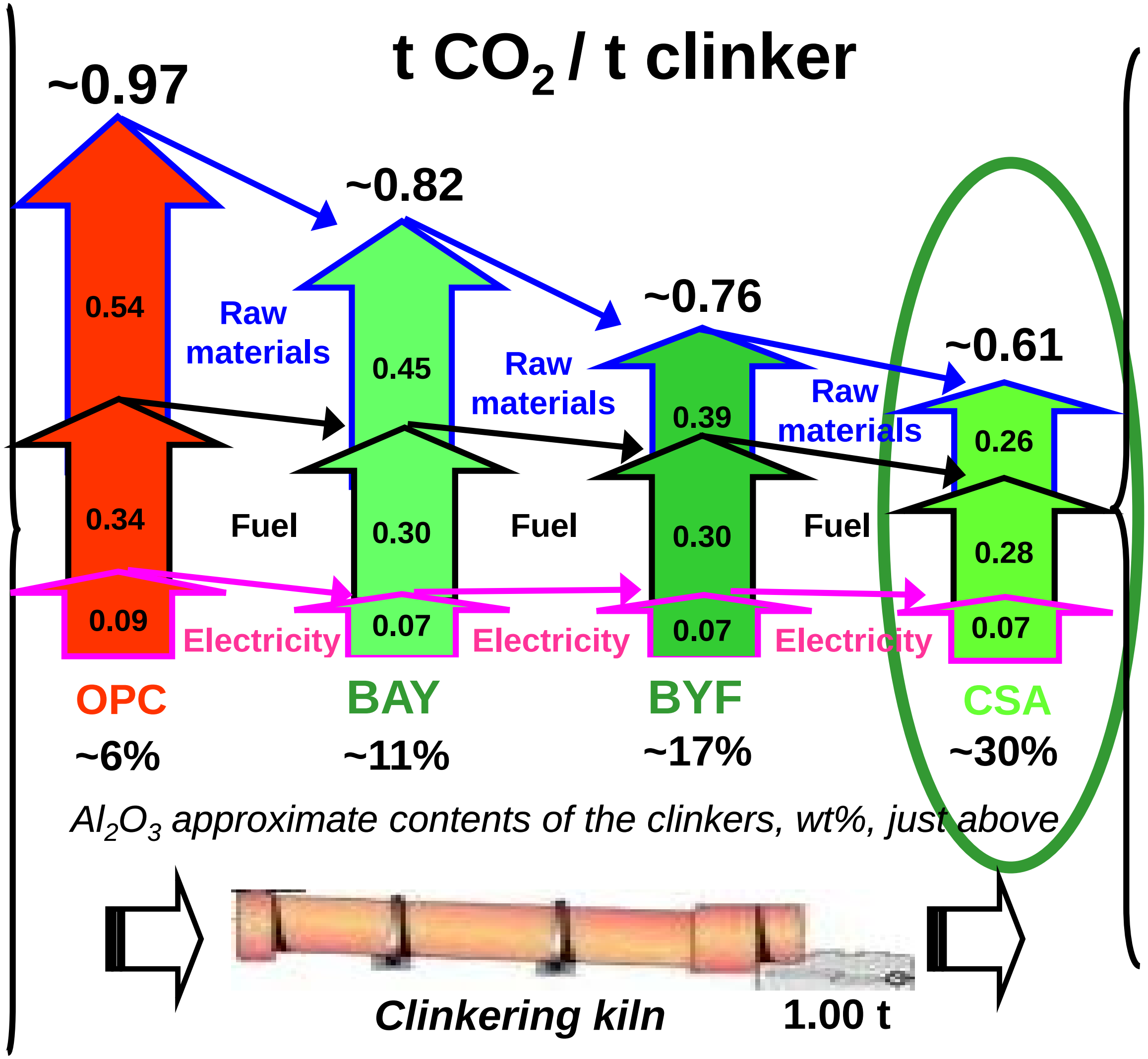

OPC clinker
Phases
65 wt% C_3S
15 wt% C_2S
10 wt% C_4AF
10 wt% C_3A

BAY clinker
Phases
55 wt% C_2S
15 wt% C_3S
15 wt% $C_4A_3\underline{S}$
15 wt% C_4AF

BYF clinker
Phases
50 wt% C_2S
30 wt% $C_4A_3\underline{S}$
20 wt% C_4AF

CSA clinker
Phases
65 wt% $C_4A_3\underline{S}$
20 wt% C_2S
6 wt% C_4AF
9 wt% $C\underline{S}$

Cement research @ UMA > eco-cements CSA

Cement & Concrete Composites 55 (2015) 53–61

Contents lists available at ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Effect of calcium sulfate source on the hydration of calcium sulfoaluminate eco-cement

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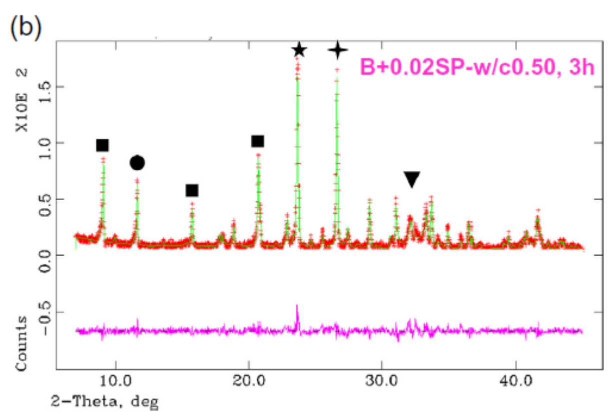


Fig. 4. (a) LXRPD diffractograms of B-w/c0.50 with 0.020 wt% SP at different hydration times (from 20 min to 3 h) measured in transmission mode. The diffractogram of the anhydrous paste is shown for the sake of comparison. (b) Diffractogram (transmission) with a Rietveld analysis of the same paste after 3 h of hydration, where the peaks of the main phases are marked.

The dissolution rate of the sulfate sources is key to control the hydration reactions.

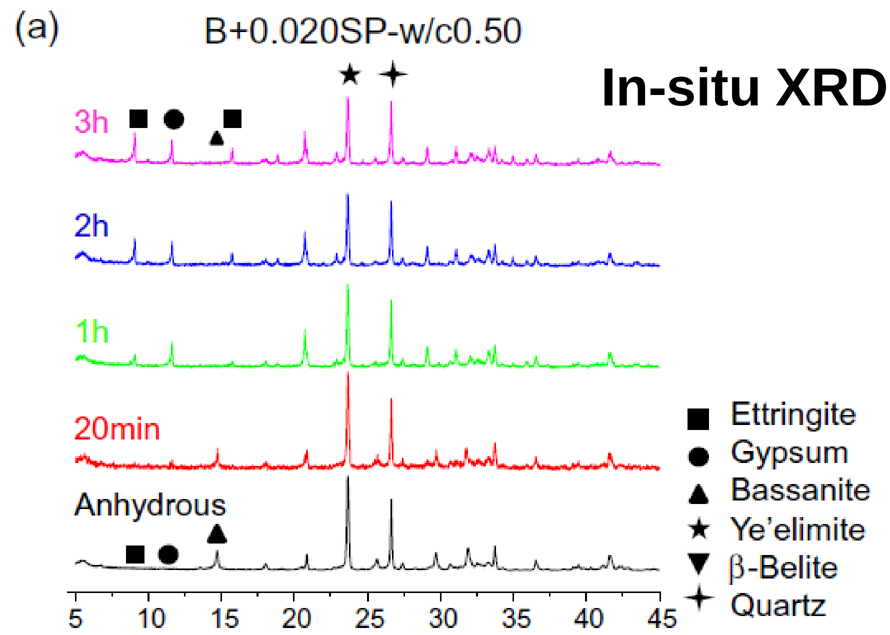


Table 2
Rietveld quantitative phase analysis results in weight percentage (including ACn and FW) for B-paste with $w/c = 0.50$ as a function of hydration time obtained by in-situ LXRPD (up to 3 h) in transmission geometry.

	0 min	20 min	1 h	2 h	3 h
$C_4A_3\bar{S}$	25.2(2)	24.7(5)	23.4(3)	20.4(3)	20.2(3)
$C\bar{S}H_2$	0.0	5.3(4)	14.3(3)	12.6(3)	12.6(3)
$C\bar{S}H_{0.5}$	13.3(2)	6.5(4)	0.0	0.0	0.0
β-C_2S	2.5(5)	2.9(9)	5.3(8)	5.2(8)	4.7(6)
Minor PHASES[a]	2.7(3)	1.2(4)	1.6(2)	1.7(2)	1.6(2)
ACn + FW	56.3	57.9	48.0	48.8	47.6
AFt	0.0	0.8(4)	7.0(3)	10.9(4)	12.9(4)
Gibbsite	0.0	0.7(4)	0.4(2)	0.4(2)	0.4(2)
SUM	100.0	100.0	100.0	100.0	100.0

[a] Overall amount of cement crystalline minor phases: $CaTiO_3$ and MgO.

Internal standard method

Cement research @ UMA > eco-cements CSA

Cement and Concrete Composites 72 (2016) 39–47

Contents lists available at ScienceDirect

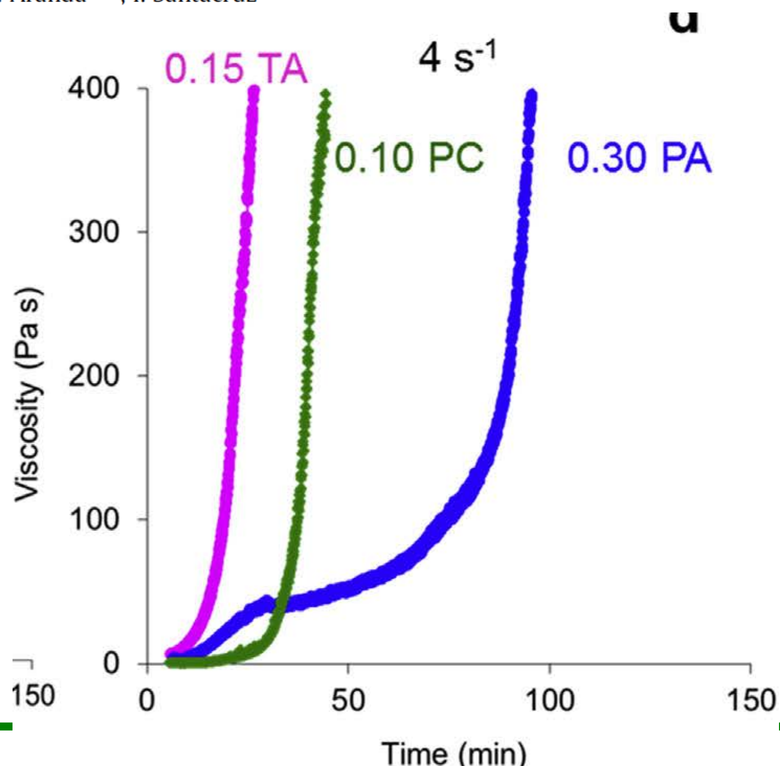
Cement and Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

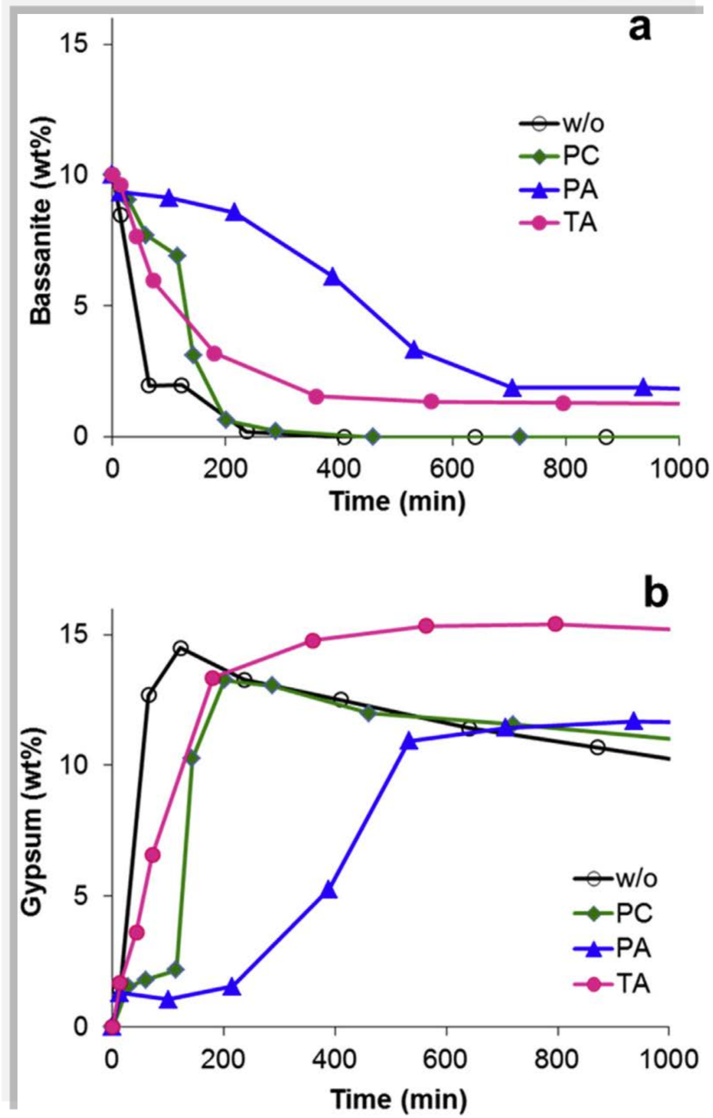
ELSEVIER

Tailored setting times with high compressive strengths in bassanite calcium sulfoaluminate eco-cements

M. García-Maté [a], D. Londono-Zuluaga [a], A.G. De la Torre [a], E.R. Losilla [a], A. Cabeza [a], M.A.G. Aranda [a, b], I. Santacruz [a, *]



Evolution of the viscosity with time (at 4 s^{-1})



Phase evolution of Bassanite (a), and Gypsum (b) (in weight percentage) with time, within the first 1000 min of hydration.

Cement research @ UMA > Hydration and XRD

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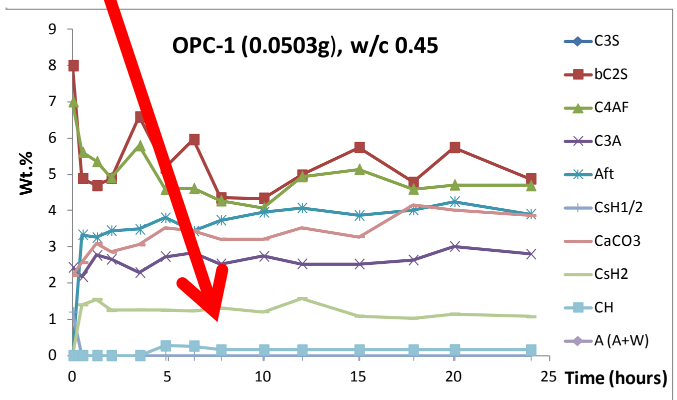
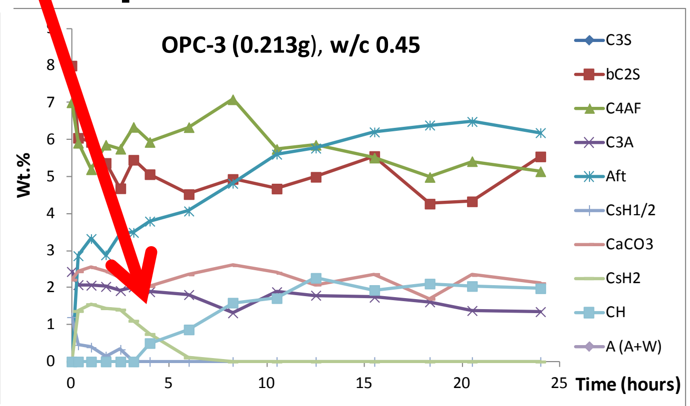
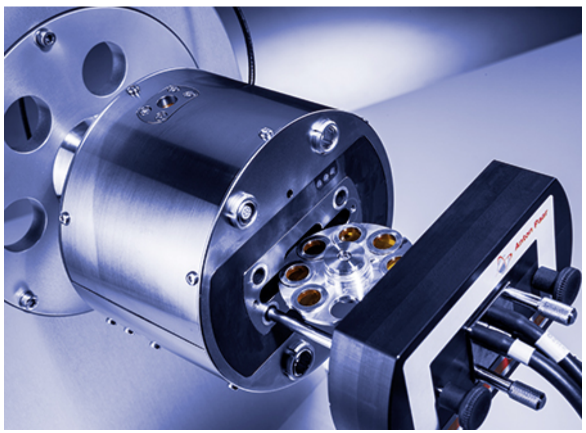
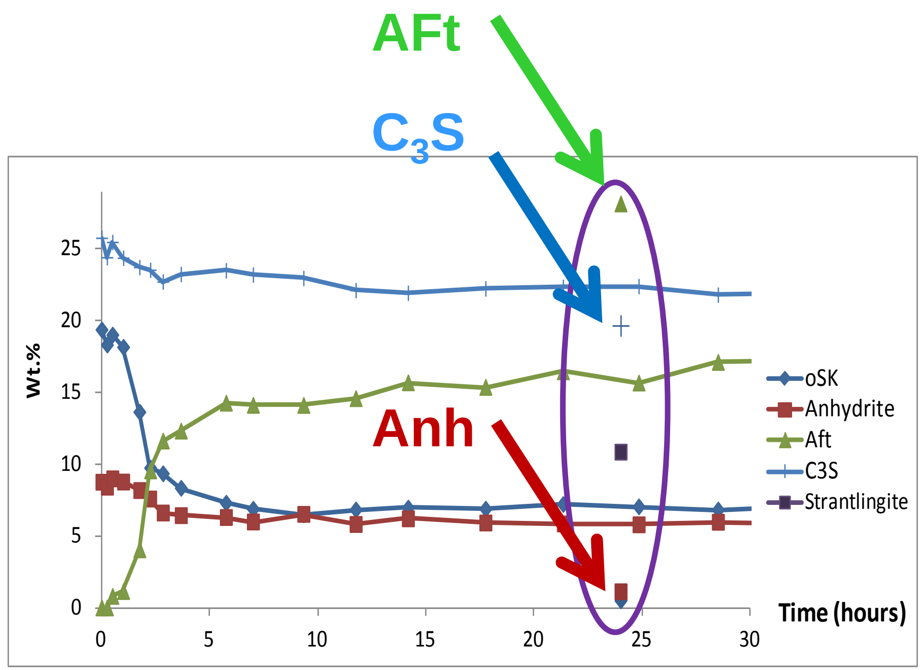
Amount of sample





D8 (Mo radiation) Transmission geometry + HUMIDITY CHAMBER + 2D DETECTOR





Possible solution

- ✓ **Sample between kapton in both sides**

Spinning (mandatory)

Cement research @ UMA > Synchrotron XRD & hydration

Construction and Building Materials 101 (2015) 818–827

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Hydration of C_4AF in the presence of other phases: A synchrotron X-ray powder diffraction study

A. Cuesta [a], I. Santacruz [a], S.G. Sanfélix [b], F. Fauth [c], M.A.G. Aranda [a,c], A.G. De la Torre [a,*]

Cement and Concrete Research 63 (2014) 127–136

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Hydration mechanisms of two polymorphs of synthetic ye'elimite

A. Cuesta [a], G. Álvarez-Pinazo [a], S.G. Sanfélix [b], I. Peral [c], M.A.G. Aranda [a,c], A.G. De la Torre [a,*]

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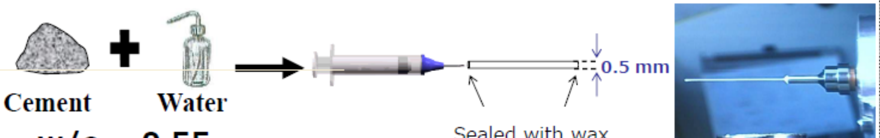
[b] Unidad Técnica de Investigación de Materiales, AIDICO, Avda. Benjamín Franklin, 17 Paterna, Valencia, Spain
[c] ALBA-CELLS Synchrotron, Carretera BP 1413, Km. 3.3, E-08290 Cerdanyola, Barcelona, Spain

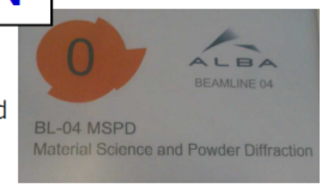
Hydration procedure



w/c = 0.55

DATA COLLECTION

October 2012
First ALBA users @ BL04 - MSPD: Materials Science and Powder Diffraction



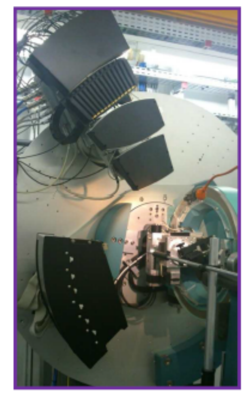
λ= 0.61984(3) Á

Debye Scherrer configuration

Capillaries were spun

Angular range 1-35º (in 2θ)

15 minutes per pattern

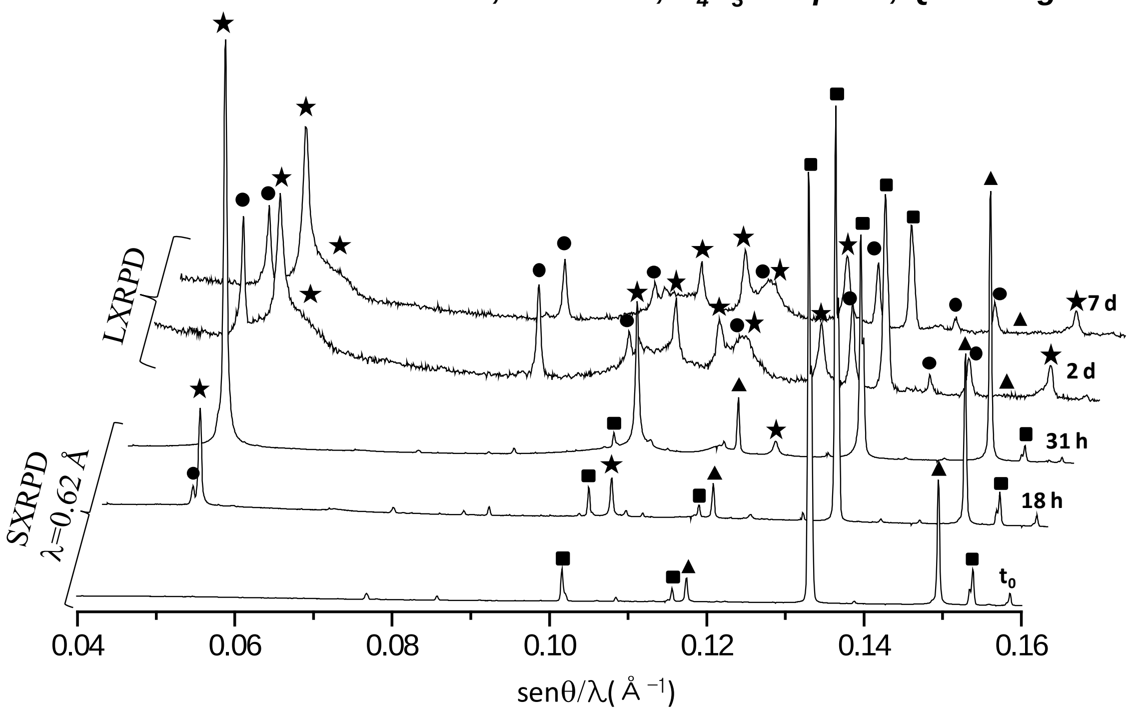


MYTHEN Detector

Cement research @ UMA > Synchrotron XRD & hydration

st-$C_4A_3\overline{S}$_1.16							
	SXRPD					***LXRPD***	
	t_0	12h	18h	24h	31h	*2d*	*7d*
st-$C_4A_3\overline{S}$	44.9(1)	43.0(1)	29.4(1)	10.3(3)	9.2(3)	*4.6(2)*	*2.9(2)*
AFt	-	-	2.3(1)	1.1(2)	1.0(2)	*9.2(3)*	*6.8(3)*
AFm	-	-	7.1(1)	22.0(2)	22.2(2)	*25.1(4)*	*25.8(5)*
ACn+FW	1.4(1)+53.7*=55.1	57.0(1)	61.2(1)	66.5(2)	67.6(2)	*44.1(5)+17=61.1*	*46.5(6)+17.9=64.4*

AFt: circle, AFm: star, C_4A_3S: square, Qz: triangle.



st-C4A3S_1.16 recorded at different hydration ages

SXRPD & LXRPD: Both strategies were able to quantify the amorphous contents, including free water. It is important to highlight that the results obtained by the internal standard method are in agreement with those obtained at later ages showing the consistence of both methodologies to follow hydration reactions with time by diffraction methods.

Cement research @ UMA > Synchrotron XRD & PDF

Cement and Concrete Research 96 (2017) 1–12

Contents lists available at ScienceDirect

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journal homepage: www.elsevier.com/locate/cemconres

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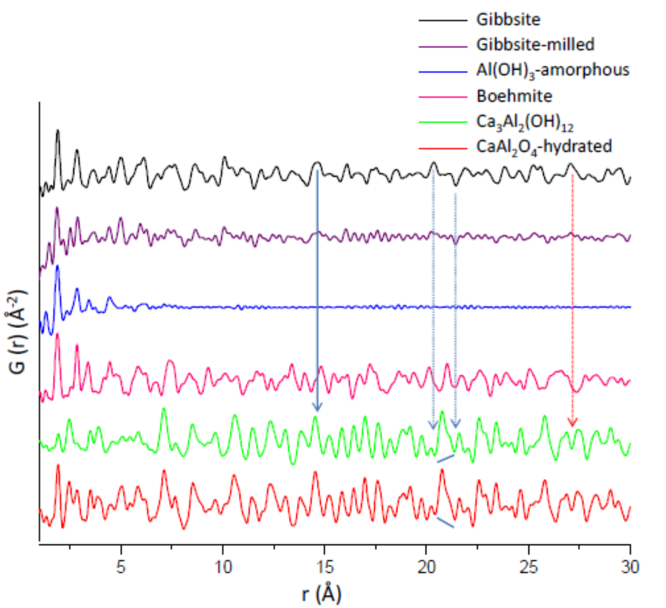
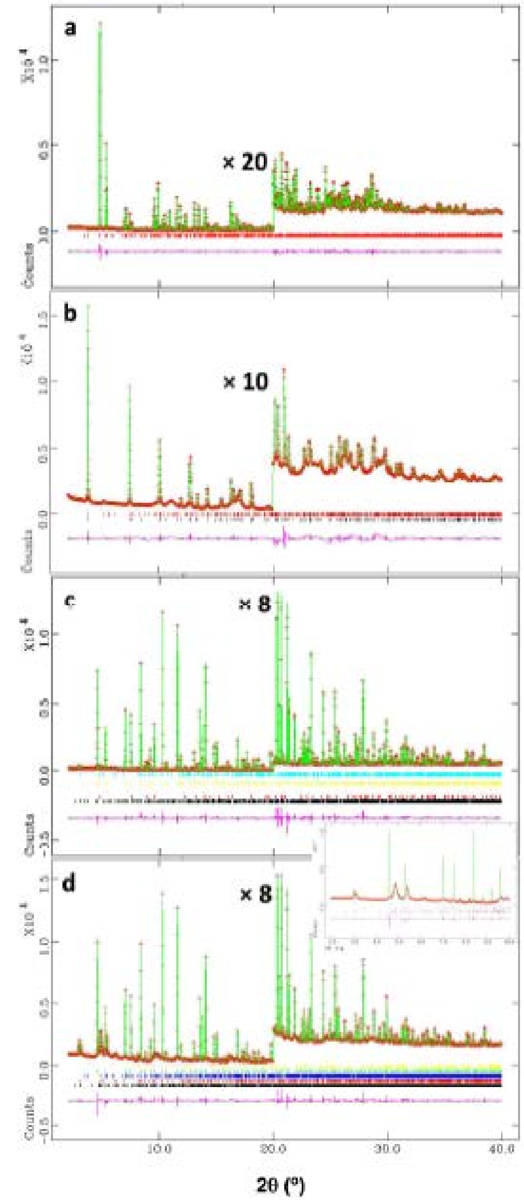


Fig. 4. Experimental PDFs for as-received commercial crystalline gibbsite, milled gibbsite, as-received commercial amorphous aluminum hydroxide, boehmite, $Ca_3Al_2(OH)_{12}$ and hydrated $CaAl_2O_4$



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X-RAY DATA SERVICES

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ELSEVIER

Aluminum hydroxide gel characterization within a calcium aluminate cement paste by combined Pair Distribution Function and Rietveld analyses

CrossMark

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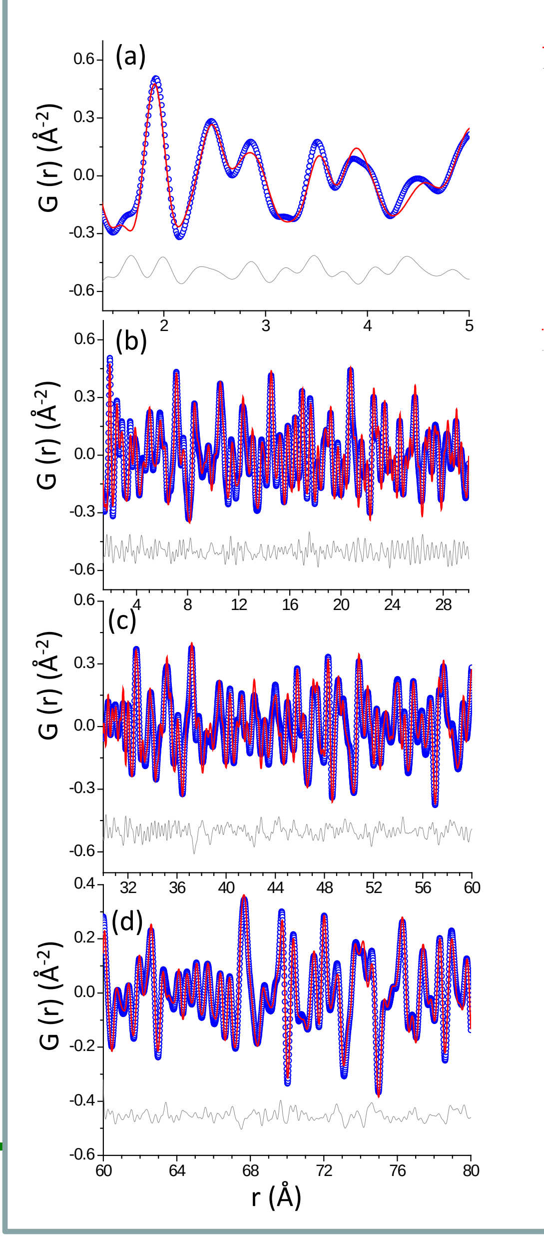
Phases	RQPA	PDF-QPA
$Ca_3Al_2(OH)_{12}$ (crystalline)	43 wt%	42.0 wt%
$Al(OH)_3 \cdot 0.1H_2O$ (nanocrystalline)	50 wt%	52.8 wt%

5 nm



Cement and Concrete Research xxx (2017) xxx-xxx

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Experimental and theoretical high pressure study of calcium hydroxyaluminate phases

A. Cuesta[a], P. Rejmak[b], A. Ayuela[c], A.G. De la Torre[d], I. Santacruz[d], L.F. Carrasco[e], C. Popescu[a], M.A.G. Aranda[a, d, *]

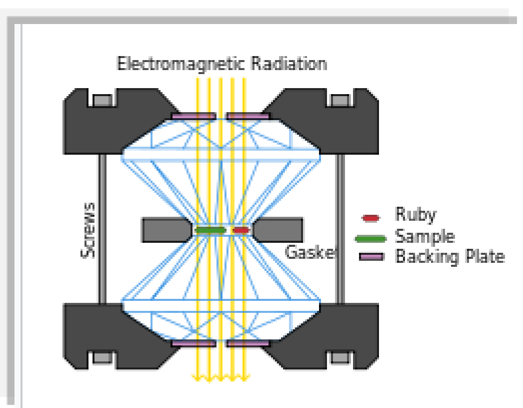

Table 1
Theoretical and experimental bulk modulus (K_0) including its derivative (K_0') for all the studied phases.

Phase	Theoretical (T)/experimental (E)	K_0 (GPa)	K_0' (GPa)
$Ca_3Al_2(OH)_{12}$	E (silicon oil)	81(2)	4.0
	E (methanol/ethanol)	76(2)	6.6(7)
	T	56.7	3.7
$Ca_3Al_{1.7}Fe_{0.3}(OH)_{12}$	E (silicon oil)	73(1)	4.0
	E (methanol/ethanol)	58(1)	4.0
	T	48.4	6.5
$Ca_4Al_2(OH)_{12}[(SO_4)\cdot 6H_2O]$	E (silicon oil)	23(1)	4.0
$Ca_6Al_2(OH)_{12}(SO_4)_3\cdot 26H_2O$	E (silicon oil)	30(3)	4.0
$Ca_4Al_2(OH)_{12}[Al_2Si_2O_4(OH)_8\cdot 6H_2O]$	E (silicon oil)[b]	27(1)	4.0
	E (silicon oil)[a]	58(6)	4.0

[a] High pressure range (1.5–5.3 GPa).

[b] Low pressure range (0–1.5 GPa).

Cement research @ UMA > Synchrotron XRD & hydration

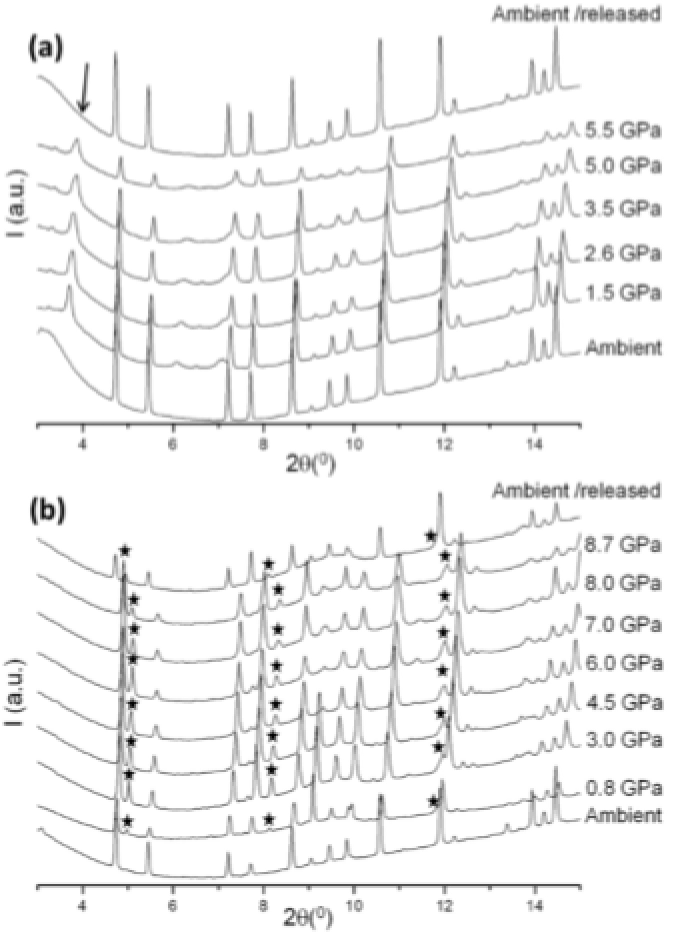

Fig. 5. Selected integrated SXRPD raw patterns, λ = 0.4246(1) Å, for $Ca_3Al_{1.7}Fe_{0.3}(OH)_{12}$ as a function of pressure. (a) Silicone oil as PTM. (b) Same sample but using ethanol/ethanol 4:1 as PTM. The peak arrowed in (a) does not arise from katoite and it may be justified by crystallization of dehydrated monocarboaluminate phase. The peaks starred in (b) denote reflections of iron/aluminum hydroxide phases.

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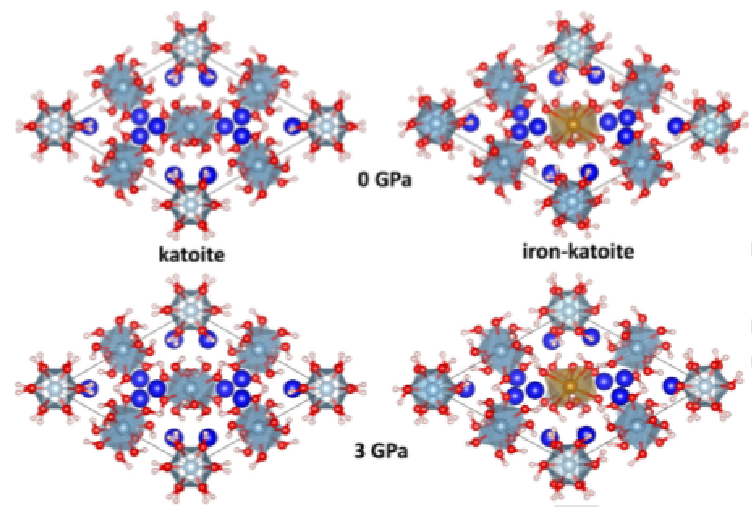


Fig. 11. View of the theoretical optimized unit cells of stoichiometric katoite (left column) and iron-containing katoite (right column) at equilibrium (upper panels) and 3.0 GPa (bottom panels), shown projected along (010) plane. Colors denote the following atoms: dark blue-Ca, light blue-Al, brown-Fe, red-O, and white-H. Ca—O bonds are omitted for the sake of clarity. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Spin off: X-Ray Data Services

Analysis

(Phase analysis, Amorphous determination)

Provide support & advise

Innovation & Knowledge transfer

(Research collaborations, Project assessment, ...)

XDS X-RAY DATA SERVICES

Training

(Data collection, Analysis interpretation, ...)

Provide collaboration to research groups

Consulting

(Protocol validation, Regulations, ...)

Provide advise

X-Ray Data Services: training activity

- **4 courses to companies with a diffractometer, (proper data collection, data analysis, validating their procedures), focus to control quality lab staff of cement factories (2) and pigment factories (2)**

- 3 courses to member of research groups @ Universities with diffractometers in their labs.

- 2 specialized courses of XRD to control quality lab staff of cement factories and cement sector.

- 1 specialized course to ceramic sector of Castellón (Spain) with 19 students.

- 1 specialized course in customs central laboratory of the tax agency of Spain.

X-Ray Data Services: data analysis activity

Data analysis activity

- **Cement (anhydrous and paste) characterization.**
- **RQPA of crystalline SiO_2 in cements.**
- **Hydration of cements: setting time.**
- **Analysis of hardened precast.**

Cement (anhydrous and paste) characterisation

The studied sample was an environmentally-friendly cement sample from Henkel.

SXRPD (BL04-MSPD, ALBA)

Data collection

λ= 0.620085(3) Å

Debye Scherrer configuration

Capillaries were spun

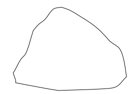
Angular range 1-35º (in 2θ)

15 minutes per pattern

MYTHEN Detector

Cement (anhydrous and paste) characterization

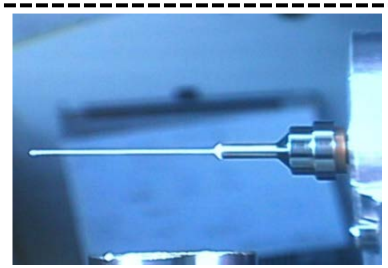
SAMPLE PREPARATION

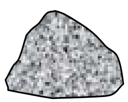
Henkel binder_H1 + 

15 wt% of Quartz (internal standard)

20 minutes →

HYDRATION PROCEDURE



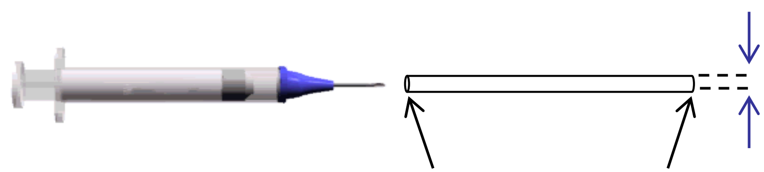


Cement

+

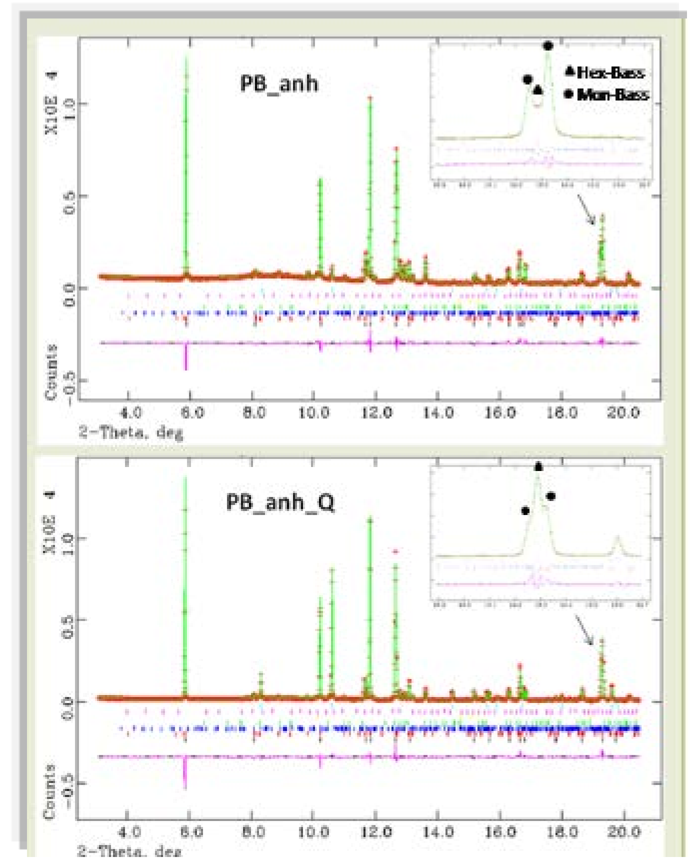
Water

→



Cement (anhydrous and paste) characterization

XDS have supported a private company in the sample preparation, data collection and data analysis in the hydration of different types of cements.



The company used these results for the understanding and development of improved cement materials.

RQPA of *Respirable Crystalline Silica (RSC)* in cements
IECA & Cement factories *are involved*

<u>Round Robin</u>

The project includes:

- Density
- Particle size distribution
- Quantification of crystalline quartz, Cristobalite and Tridymite

XDS X-RAY DATA SERVICES **were hired as external laboratory B**

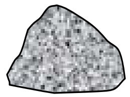
RQPA of cements including of <u>crystalline silica</u> (quartz, Cristobalite and Tridymite)

Hydration of cements: setting time

X-RAY DATA SERVICES

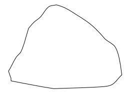
UNIVERSIDAD DE MÁLAGA

SAMPLE PREPARATION



Cement

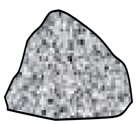
+



20 wt% of Quartz (internal standard)

20 minutes →

HYDRATION PROCEDURE: in-situ XRD

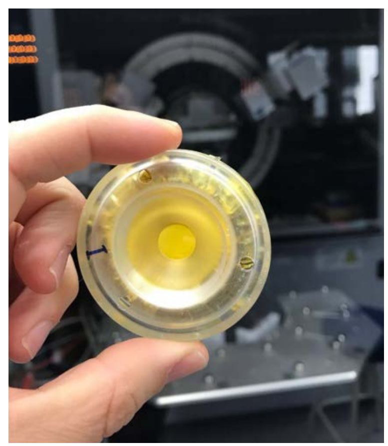


Cement

+

Water

w/c = 0.45



D8ADVANCE

First diffractometer installed in Europe

Mo $K\alpha_1$ radiation

Hydration of cements: setting time

Example of RQPA (included Amorphous and free water) of OPC cement by in-situ XRD

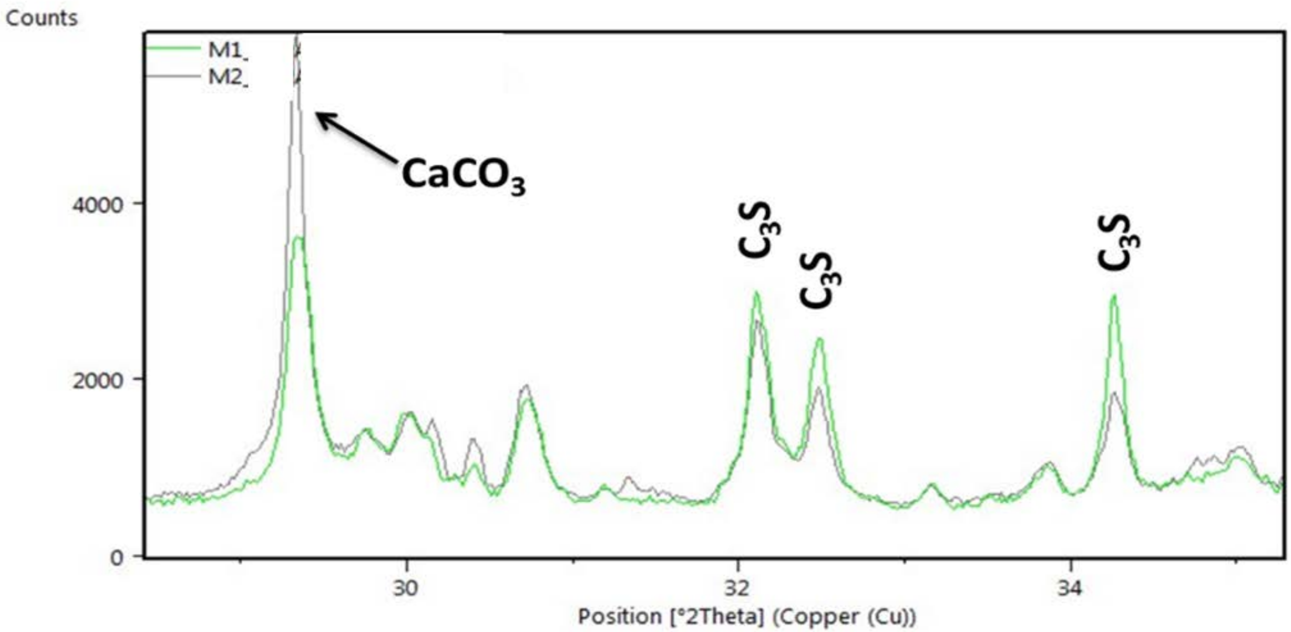
Fases	t0	5min	1h	2h	3h	4h	5h	6h
C_3S (silicato tricálcico)	36.7	36.5	37.1	37.0	34.1	29.1	26.4	24.5
β-C_2S (silicato dicálcico)	6.6	5.3	5.5	5.9	4.7	5.3	6.1	7.1
C_4AF (ferritoaluminato tetracálcico)	**4.0**	**4.0**	**3.9**	**3.9**	**4.0**	**4.1**	**3.8**	**3.5**
C_3A (aluminato tricálcico)	**4.3**	**3.5**	**3.4**	**3.7**	**3.4**	**3.4**	**3.2**	**2.7**
C (cal libre)	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
M (periclasa)	0.2	0.1	0.1	0.2	0.2	0.3	0.2	0.3
CsH_2 (yeso)	**0.7**	**1.3**	**1.1**	**1.1**	**0.9**	**0.5**	**0.8**	**0.9**
$CsH_{0.5}$ (basanita)	**0.4**	**0.2**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**	**0.0**
Cc (calcita)	3.1	3.1	3.5	3.7	3.7	3.7	3.2	3.9
KC_2s_3 (langbeinita)	0.3	0.3	0.4	0.5	0.5	0.5	0.3	0.2
Ks (arcanita)	0.4	0.3	0.4	0.4	0.2	0.4	0.3	0.3
K_3Ns_4 (aftitalita)	0.1	0.5	0.5	0.4	0.1	0.2	0.2	0.4
CH (portlandita)	0.8	0.8	0.8	0.8	1.9	3.2	4.3	4.9
$C_6As_3H_{32}$ (Etringita)		3.8	3.9	4.0	4.9	5.6	6.9	7.1
ACn + FW	*42.2*	*40.3*	*39.3*	*38.5*	*41.5*	*43.5*	*44.3*	*44.3*

The different behavior in hydration can be determined by analysing a normal and a problematic sample.

Problem: Analysis of hardened precast concrete

Sample M1: surface area of a precast concrete exposed to the sun.

Sample M2: hydrated sample M1 (48 hours and dried with acetone).



C_3S peaks indicate unreacted cement in the surface area.

Hydrated sample (grey line) shows a slight decrease of C_3S, indicating that there was hydration.

XRD and Cements: from research to control quality

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