Empa GEMS workshop 2014

1.2 Tutorial 2

Single system - Hydration of OPC

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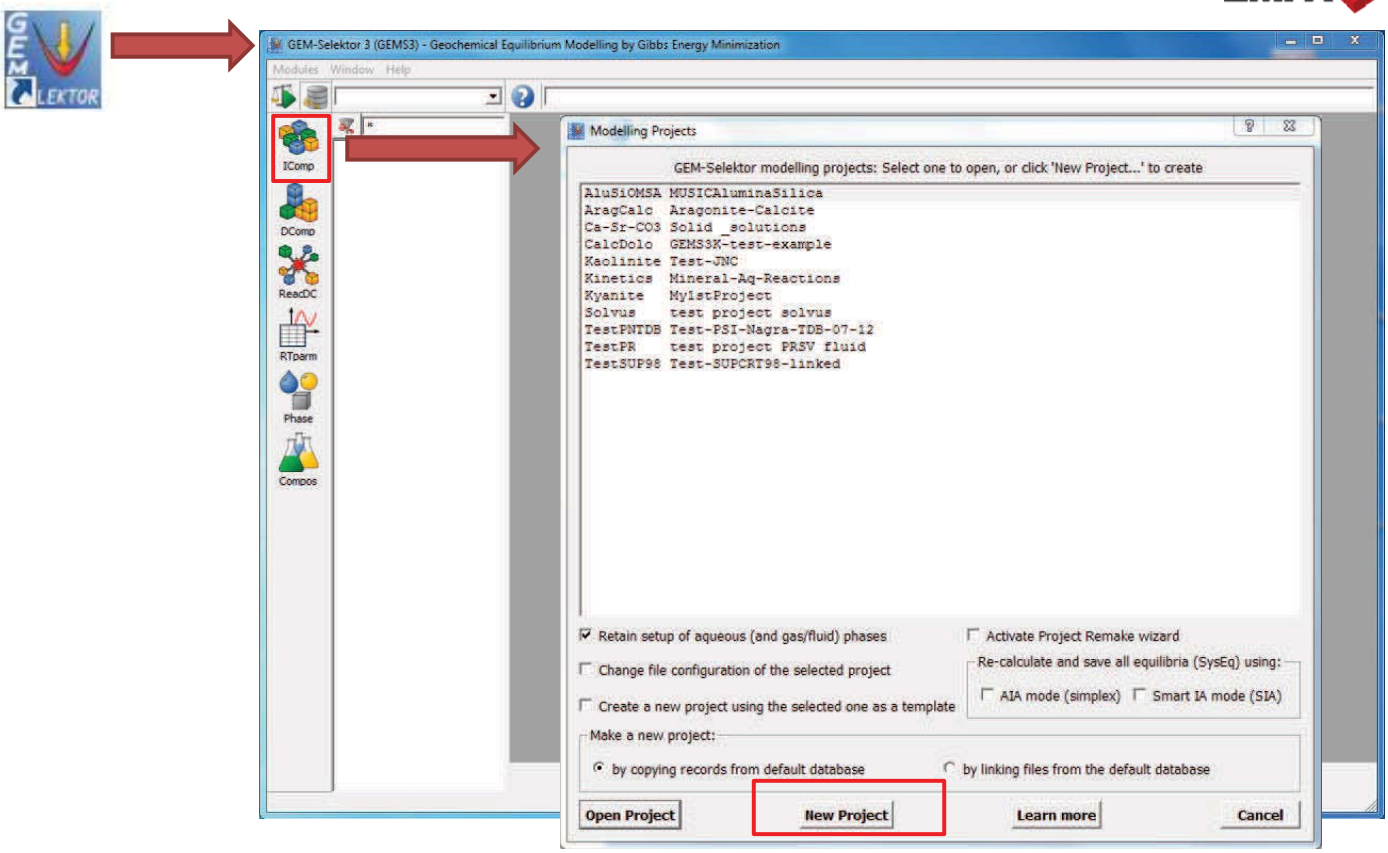
GEMS V.3.2 2482.930, CEMDATA14 (version April 14, 2014)

Tutorial 2 – OPC hydration

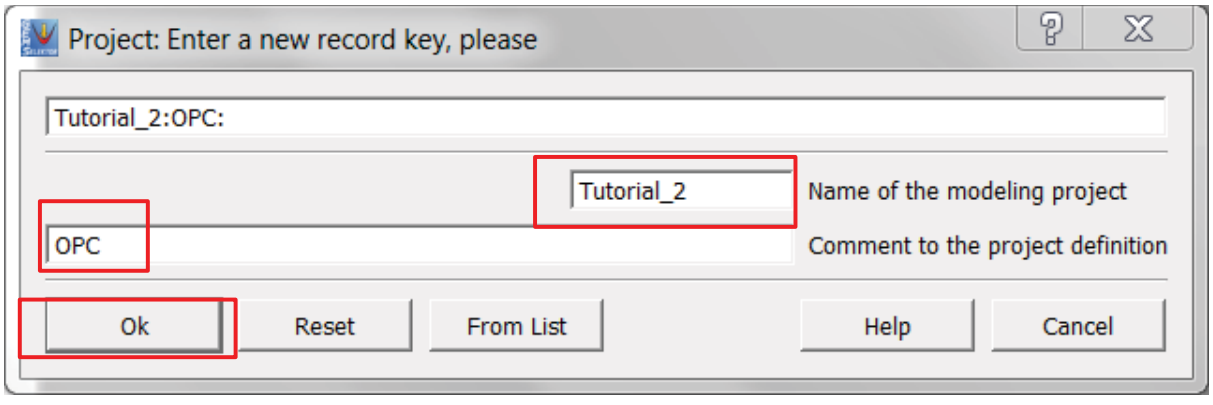
Hydration of a model cement composition

- The example will show the principle possibility to apply GEMS to simulate cement hydration.
- A simplified cement composition is used and 100% hydration assumed (other assumptions are possible).
- The impact of limestone on the predicted phase assemblage will be shown.

Hydration of OPC – project setup

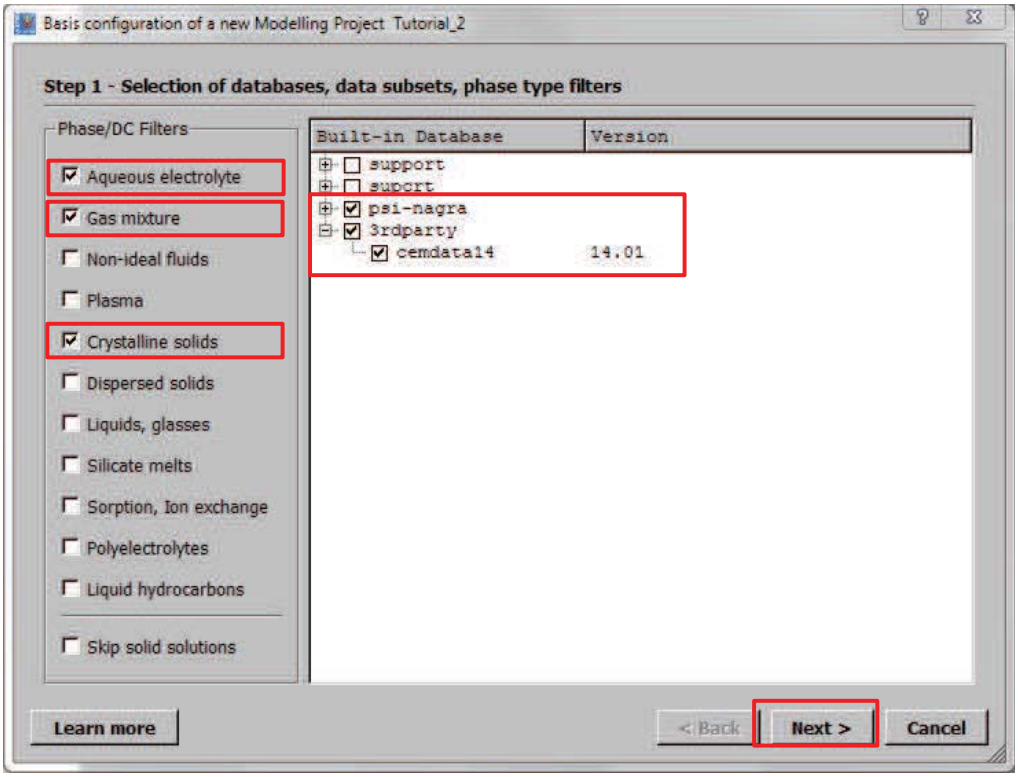


Hydration of OPC – project setup



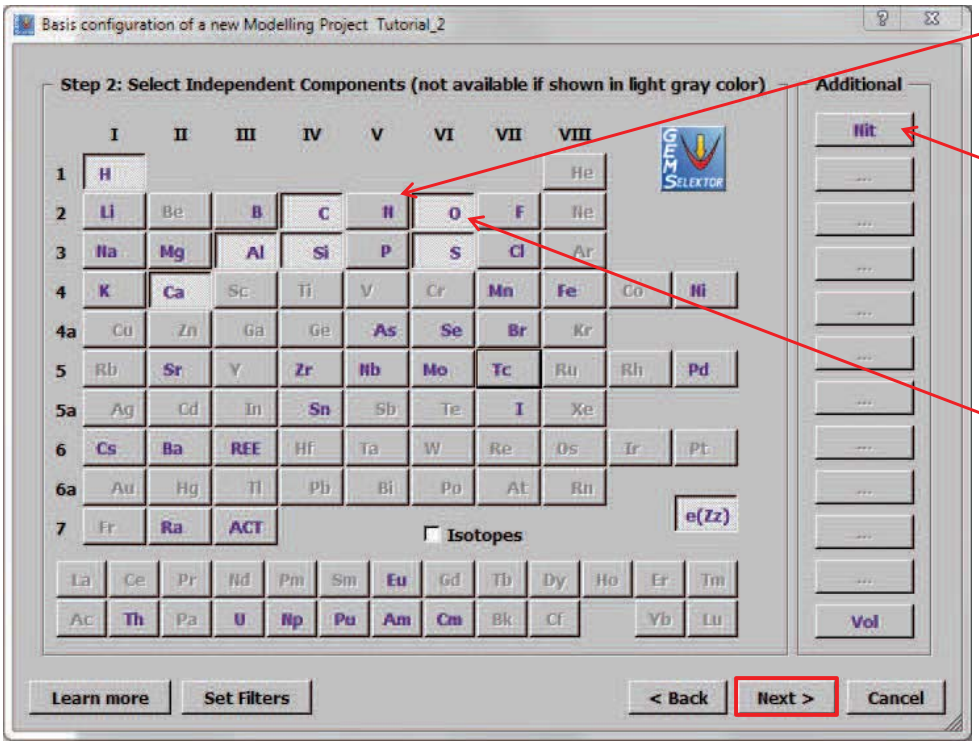
Hydration of OPC – project setup

Select CEMDATA14 (3rd party data base)



Hydration of OPC – project setup

Modified project setup including elements necessary to model cement hydration (as we use a model cement we neglect Na, K, Mg and Fe for the moment)



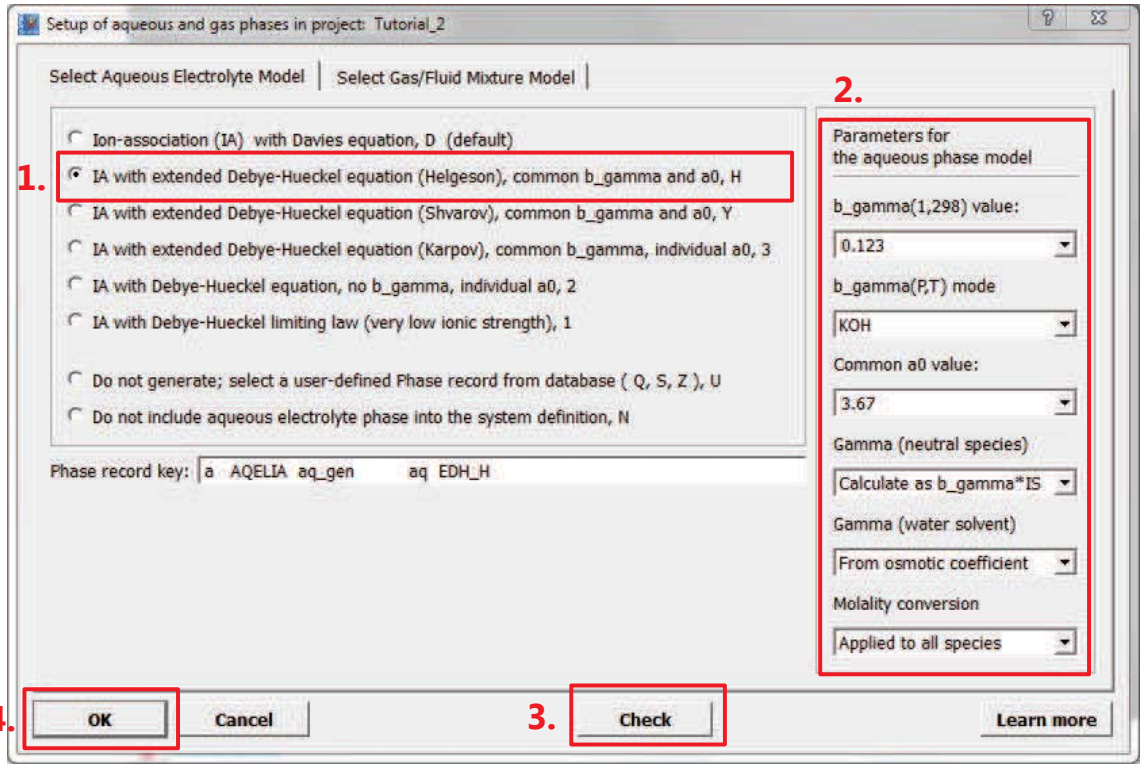
„reactive N“ (don‘t use here)

Air: O_2, N_2, etc. (no reducing conditions, N_2 not reactive)

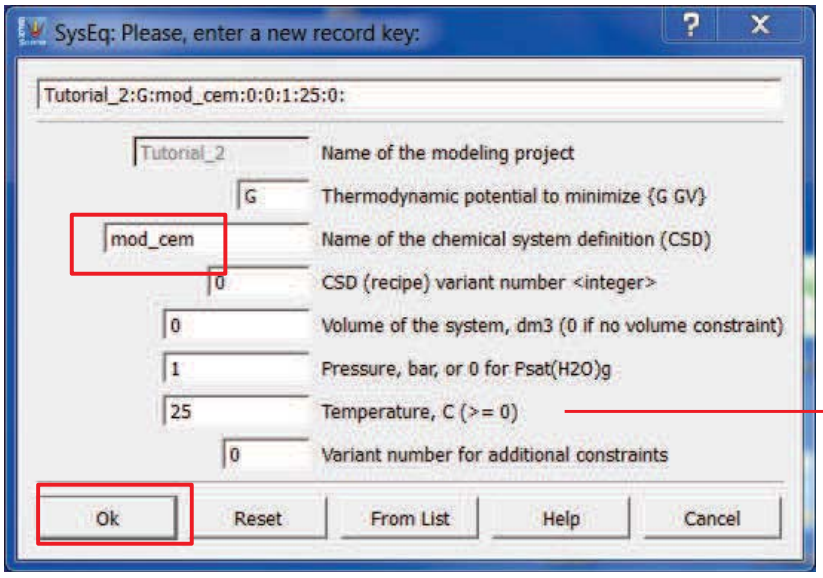
we can also use just oxygen (O_2)

Hydration of OPC – project setup

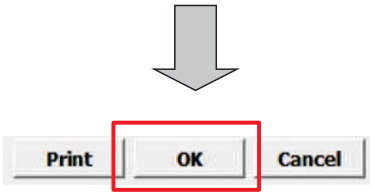
Select model for calculation of the aqueous phase



Hydration of OPC – project setup

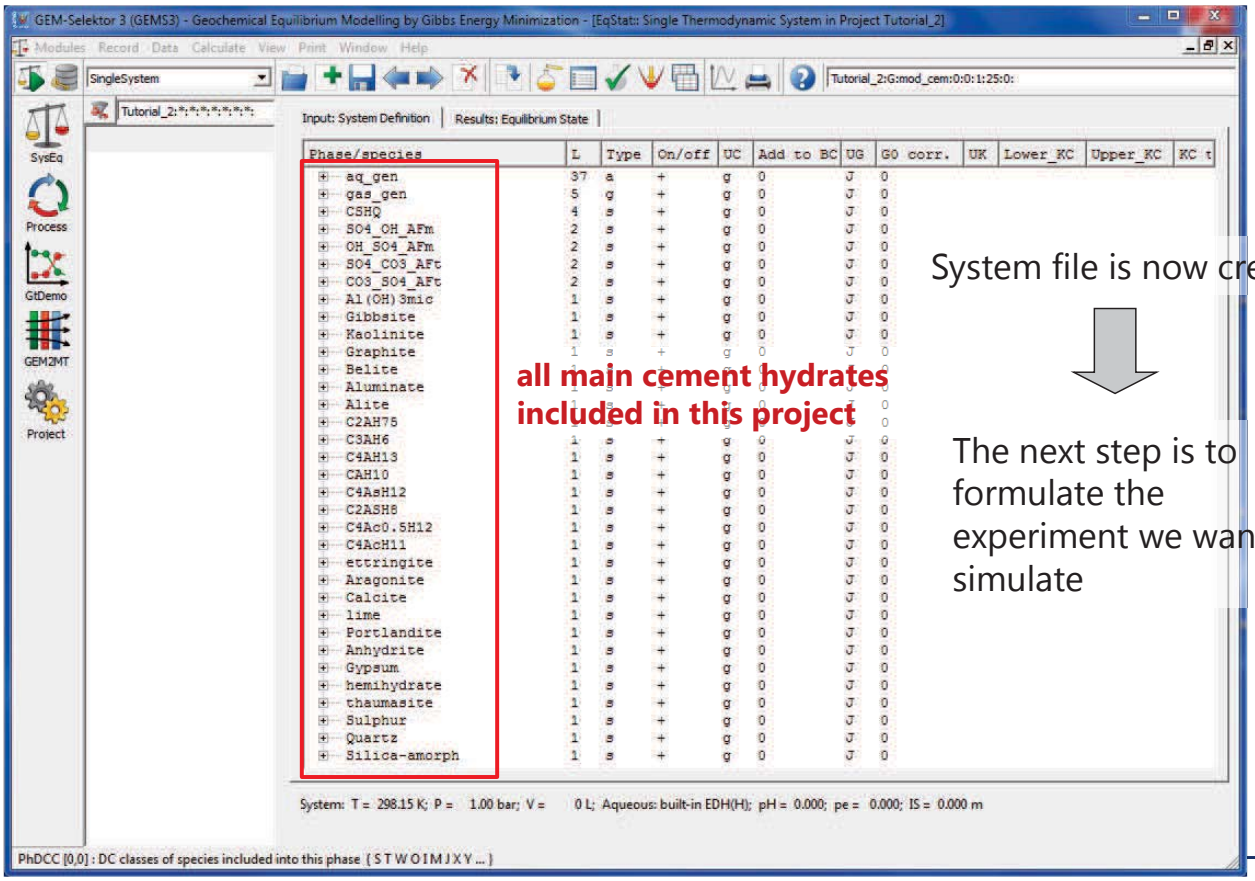


We leave temperature at 25°C

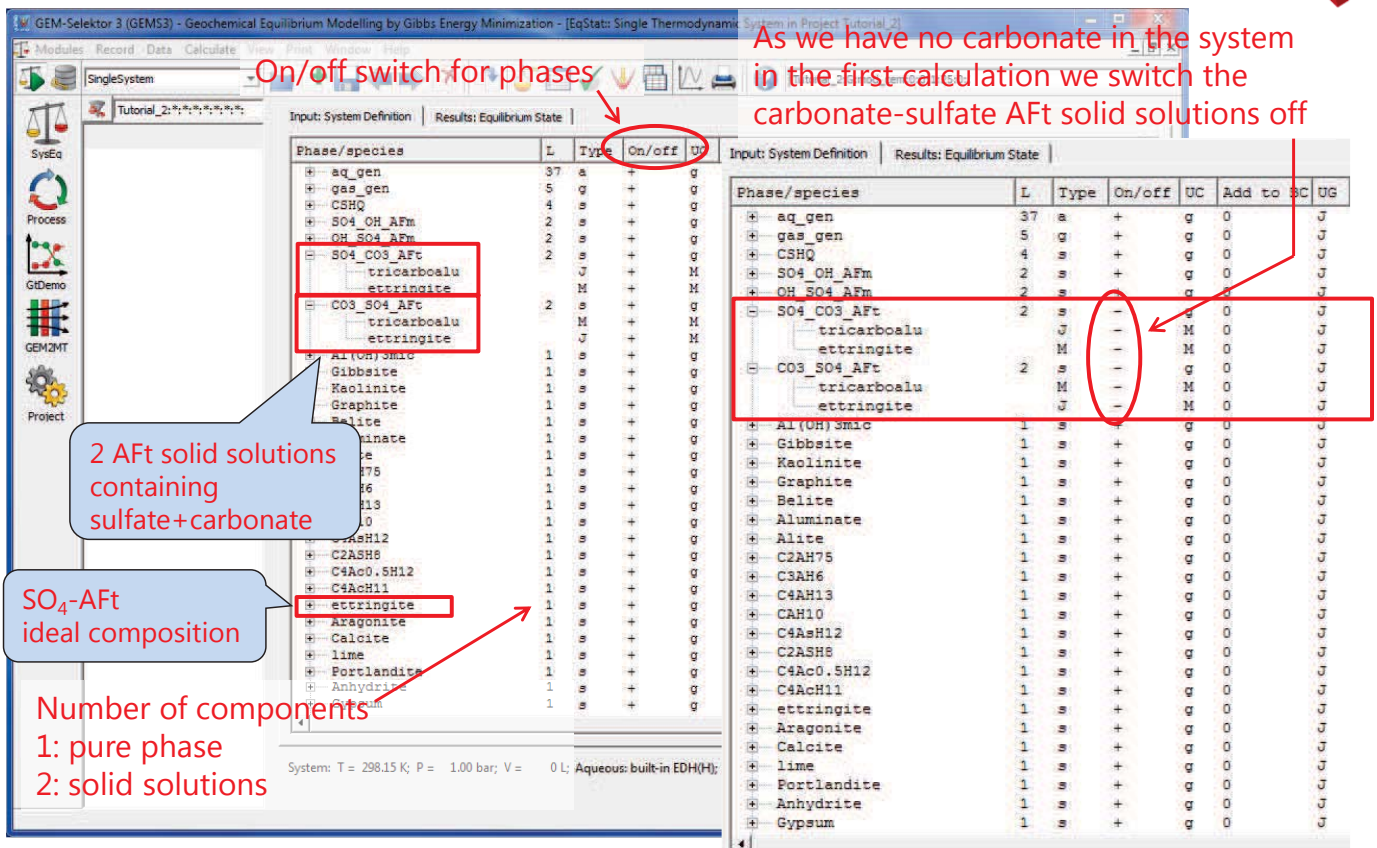


Press OK again to leave the recipe wizard. We will use it later, but first some more explanations and definitions are needed.

Hydration of OPC – system creation



Hydration of OPC – system creation



Hydration of OPC – system creation

C-S-H is modeled as a solid solution of 4 different species:

Input: System Definition | Results: Equilibrium State

Phase/species	L	Type	On/off	UC	Add to BC	UG	G0 corr.	UK	Lower_KC	Upper_KC	KC
aq_gen	37	a	+	g	0	J	0				
gas_gen	5	g	+	g	0	J	0				
CSHQ	4	s	+	g	0	J	0				
CSH-JenD		I	+	M	0	J	0	M	0	1e+006	B
CSH-JenH		I	+	M	0	J	0	M	0	1e+006	B
CSH-TobD		I	+	M	0	J	0	M	0	1e+006	B
CSH-TobH		I	+	M	0	J	0	M	0	1e+006	B

CSH-JenD = $(CaO)_{1.5}\cdot(SiO_2)_{0.6667}\cdot(H_2O)_{2.5}$

CSH-JenH = $(CaO)_{1.333}\cdot(SiO_2)_1\cdot(H_2O)_{2.1667}$

CSH-TobD = $[(CaO)_{1.25}\cdot(SiO_2)_1\cdot(H_2O)_{2.75}]_{0.6667}$

CSH-TobH = $(CaO)_{0.6667}\cdot(SiO_2)_1\cdot(H_2O)_{1.5}$

For more detailed information see: Kulik D.A., Cem. Concr. Res. 41 (2011), 477.

Hydration of OPC – system creation

Experimental problem part 1:

Hydration of a model cement

CaO	70.6 wt.-%
SiO_2	22.0 wt.-%
Al_2O_3	4.5 wt.-%
SO_3	2.9 wt.-%
w/c	*0.50*

Assumption: 100% hydration

Bogue calculation

C_3S	81.37 wt.-%
C_2S	1.70 wt.-%
C_3A	11.93 wt.-%
$CaSO_4$	5.00 wt.-%

React 100 g of the model cement with 50 g water (add 1 g O_2)

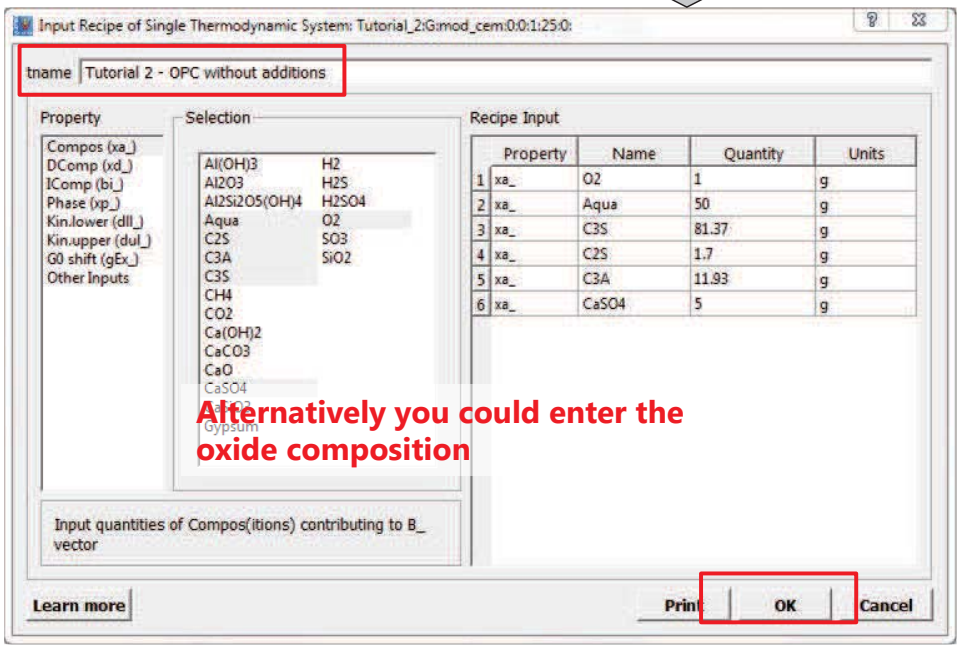
> Equilibrium speciation

Hydration of OPC – system creation

Experimental conditions: React 100 g OPC with 50 g water (add 1 g O_2)

Use recipe wizard

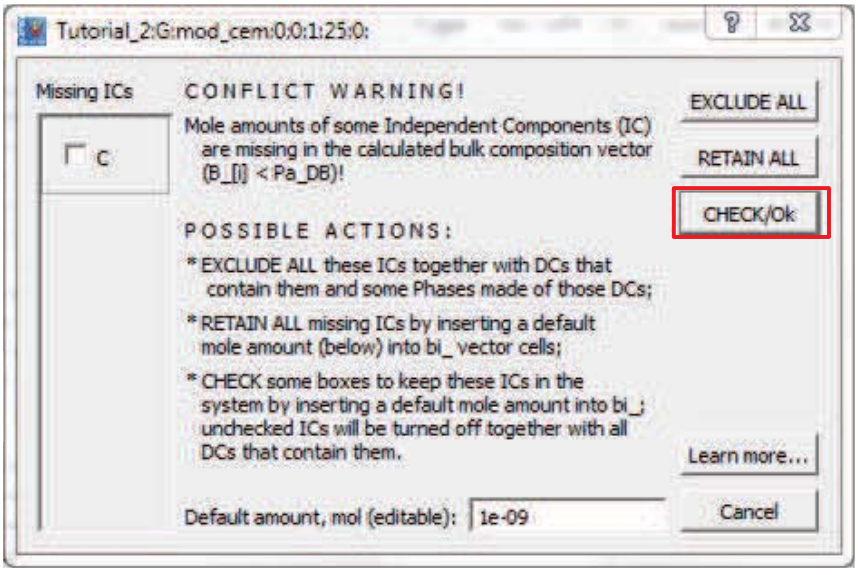
Enter phase composition



Hydration of OPC – system creation

Experimental conditions: React 100 g OPC with 50 g water (add 1 g O_2)

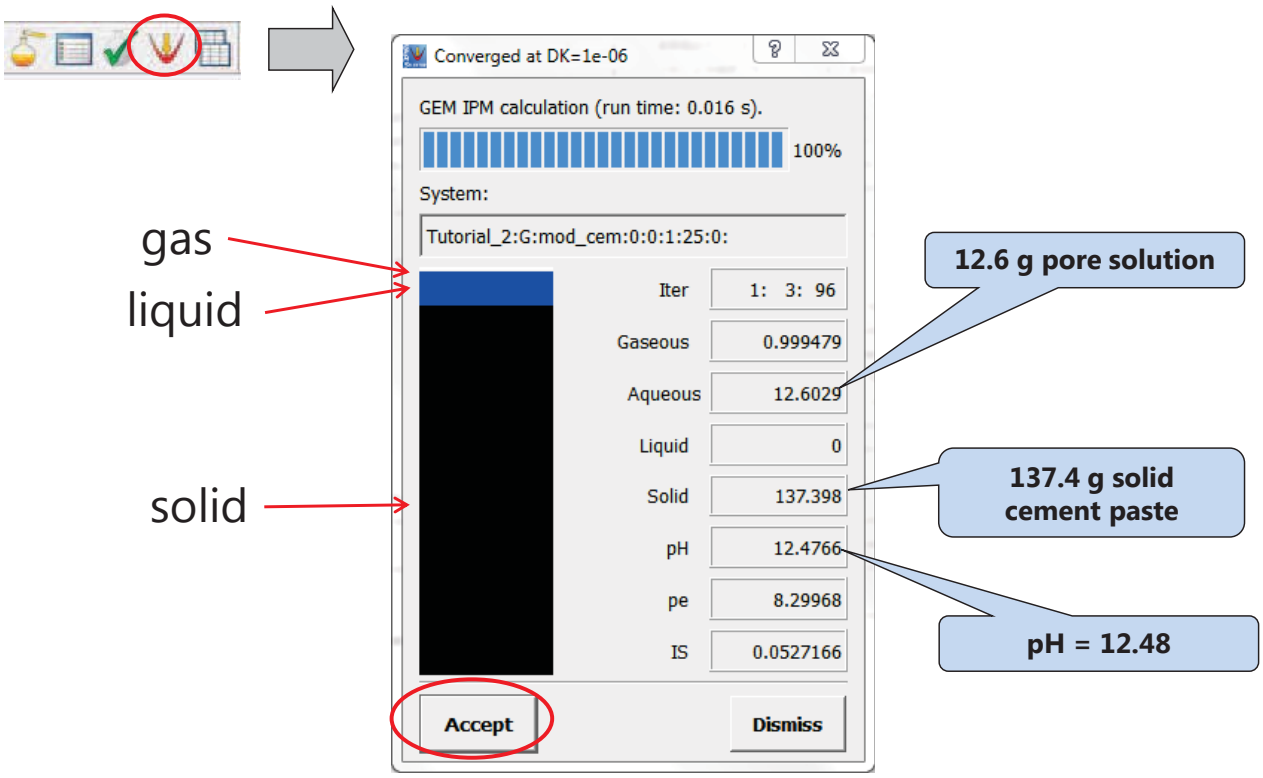
Check system definition



We exclude carbonate in this example

Hydration of OPC – calculation of equilibrium

Experimental conditions: React 100 g OPC with 50 g water (add 1 g O_2)



Hydration of OPC – results

Predicted stable phase assemblage

Input: System Definition | Results: Equilibrium State

Phase/species	L	T	Amount (mol)	logSI/Activity	Concentration	Activity coeff.
a aq_gen	31	a	0.69920713	-7.238e-10		
g gas_gen	3	g	0.031234875	4.938e-12		
s CSHQ	4	s	0.48097803	1.812e-09		
CSH-JenD		I	0.19647065	0.408482	0.40848155	1
CSH-JenH		I	0.13034662	0.271003	0.27100327	1
CSH-TobD		I	0.14771528	0.307114	0.30711441	1
CSH-TobH		I	0.0064454787	0.0134008	0.013400776	1
s SO4_OH_AFm	2	s	0.044152586	4.151e-09		
C4AH13		J	0.0074266998	0.109027	0.16820532	0.648[illegible]
monosulphate		M	0.036725886	0.709781	0.83179468	0.85331218
s OH_SO4_AFm	2	s	3.563024e-007	4.151e-09		
s Al(OH)3mic	1	s	0	-1.91		
s Gibbsite	1	s	0	-1.457		
s Kaolinite	1	s	0	-12.41		
s Belite	1	s	0	-1.754		
s Aluminate	1	s	0	-35.77		
s Alite	1	s	0	-13.79		
s C2AH75	1	s	0	-1.762		
s C3AH6	1	s	0	-0.2615		
s C4AH13	1	s	0	-0.9625		
s CAH10	1	s	0	-2.762		
s C4AsH12	1	s	0	-0.1489		
s C2ASH8	1	s	0	-0.4563		
s ettringite	1	s	0	-1.805		
s lime	1	s	0	-9.776		
s Portlandite	1	s	0.48533506	-1.575e-09		
s Anhydrite	1	s	0	-4.289		
s Gypsum	1	s	0	-4.066		
s hemihydrate	1	s	0	-5.055		
s Sulphur	1	s	0	-120.3		
s Quartz	1	s	0	-5.041		
s Silica-amorph	1	s	0	-6.073		

System: T = 298.15 K; P = 1.00 bar; V = 0.8495 L; Aqueous: built-in EDH(H); pH = 12.477; pe = 8.300; IS = 0.053 m

CSH-JenD = $(CaO)_{1.5}\cdot(SiO_2)_{0.6667}\cdot(H_2O)_{2.5}$
CSH-JenH = $(CaO)_{1.333}\cdot(SiO_2)_1\cdot(H_2O)_{2.1667}$
CSH-TobD = $[(CaO)_{1.25}\cdot(SiO_2)_1\cdot(H_2O)_{2.75}]_{0.6667}$
CSH-TobH = $(CaO)_{0.6667}\cdot(SiO_2)_1\cdot(H_2O)_{1.5}$

C-S-H solid solution
1.63 CaO · SiO_2 · 2.88 H_2O

SO_4-AFm solid solution

0.83 mol-% SO_4-AFm
0.17 mol-% OH-AFm

=> $Ca_4Al_2(OH)_{12.34}(SO_4)_{0.83}\cdot 6H_2O$

Hydration of OPC – results

Assessment of calculations

Predicted phase assemblage *(hydr. cement paste):*

- C-S-H (solid solution)
- SO_4-AFm (solid solution)
- Portlandite

Total solid volume:

33.13+13.38+16.05

= 62.56 cm³

EqIC | EqPh | EqDC | EqSurf

Tutorial 2 – OPC without additions

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mC-S-H = 74.41 g

$m_{pore\ sol.}$ = 12.60 g

$V_{pore\ sol.}$ = 12.62 cm³

$V_{C\text{-}S\text{-}H.}$ = 33.13 cm³

$V_{SO4\text{-}AFm.}$ = 13.38 cm³

$m_{SO4\text{-}AFm}$ = 27.03 g

$V_{Portlandite}$ = 16.05 cm³

$m_{Portlandite}$ = 35.96 g

		PHnam	L1	Xa	Fa	phVol	pVM
0	a	aq_gen	31		010	12.61 18	12.602856
1	g	gas_gen	3		012	774.30355	0.99947852
2	s	CSHQ	4		009	33.130544	74.412635
3	s	SO4_OH_AFm			009	13.384168	27.025028
4	s	OH_SO4_AFm			022e-009	0.00010800812	0.00 02180877
5	s	SO4_CO3_AFt			-1.88 06	0	0
6	s	CO3_SO4_AFt	1	0	04606	0	0
7	s	Al(OH)3mic	1	0	2.9104438		
8	s	Gibbsite	1	0	72937		
9	s	Kaolinite	1	0	11261		
10	s	Belite	1	0	-1.7543898	0	0
11	s	Aluminate	1	0	-35.773964	0	0
12	s	Alite	1	0	-13.792543	0	0
13	s	C2AH75	1	0	-1.7616159	0	0
14	s	C3AH6	1	0	-0.26150595	0	0
15	s	C4AH13	1	0	-0.962464	0	0
16	s	CAH10	1	0	-2.7618551	0	0
17	s	C4AsH12	1	0	-0.14887592		
18	s	C2ASH8	1				
19	s	ettringite	1			0	0
20	s	lime	1	0	514	0	0
21	s	Portlandite	1	0.48533506	-1.5750415e-009	16.045177	35.959785
22	s	Anhydrite	1	0	-4.2890741	0	0
23	s	Gypsum	1	0	-4.065956	0	0

Hydration of OPC – results

Assessment of calculations

Aqueous phase composition (speciation, acivities and activity coefficients) →

EqIC | EqPh | EqDC | EqSurf | EqGen | 08/04/2014, 16:45

Tutorial 2 – OPC without additions

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total conc [mol] | lg (activity) | activity coeff. | molality (mol/kg)

	DCnam	x	lga	gamma	my
0	Al(SO4)+	0	-38.724304	0.82138317	0
1	Al(SO4)2-	0	-38.217918	0.82138317	0
2	Al+3	6.1654384e-033	-31.130816	0.15101216	4.8997336e-031
3	AlO+	2.5912107e-019	-16.771743	0.82138317	2.0592602e-017
4	AlO2-	1.2062931e-006	-4.1037929	0.82138317	9.5865278e-005
5	AlO2H@	9.1118271e-013	-10.133699	1.0150502	7.2412572e-011
6	AlOH+2	7.0715173e-026	-23.611558	0.43523178	5.6198032e-024
7	AlHSiO3+2	4.0727468e-032	-29.851184	0.43523178	3.2366513e-030
8	AlSiO5-3	2.3618467e-012	-10.547528	0.15101216	1.8769825e-010
9	Ca(SO4)@	5.5818969e-009	-6.3465221	1.0150502	4.4359875e-007
10	Ca+2	0.00020326399	-2.1530105	0.43523178	0.016153586
11	CaOH+	5.353897e-005	-2.4565757	0.82138317	0.0042547938
12	Ca(HSiO3)+	1.2939784e-009	-7.0733187	0.82138317	1.0283372e-007
13	CaSiO3@	3.6858892e-007	-4.5267617	1.0150502	2.9292118e-005
14	HSiO3-	1.161123e-008	-6.1203675	0.82138317	9.227557e-007
15	Si4O10-4	1.0549231e-022	-21.541151	0.034309925	8.3835758e-021
16	SiO2@	2.0252543e-011	-8.7868244	1.0150502	1.6094892e-009
17	SiO3-2	3.0712829e-009	-6.9737511	0.43523178	2.4407783e-007
18	H2@	0	-44.658521	1.0150502	0
19	O2@	1.629693e-005	-2.8811982	1.0150502	0.0012951328
20	S2O3-2	0	-166.13589	0.43523178	0
21	HSO3-	0	-56.699599	0.82138317	0
22	SO3-2	0	-51.442988	0.43523178	0
23	HSO4-	1.5950289e-019	-16.982477	0.82138317	1.2675849e-017
24	SO4-2	9.2783654e-009	-6.4935994	0.43523178	7.3736068e-007

Aqueous phase composition (total molalities)

↓

EqIC | EqPh | EqDC | EqSurf | EqGen

m t	ICnam
9.5865538e-005	Al …
0.020438218	Ca …
0.040719136	H …
0.04359652	O …
1.1809594e-006	S …
3.0563583e-005	Si …
7.4692972e-018	Zz …

Hydration of OPC without addition – summary

Hydration of a model cement composition – summary

- possibility to apply GEMS to simulate cement hydration with metastability constraints (e.g. siliceous hydrogarnet suppressed – see appendix)
- additional iron and alkalis present in real "OPC".
  Thus to improve accuracy of the calculation one has to consider:
  - Additional iron containing phases
    (Moeschner et al. Geoch. Cosm. Acta 2007, Dilnesa et al. CCR 2014a+b included in the cement database)
  - Sorption of alkalis on C-S-H
    (*experimental data:* Hong and Glasser CCR 1999)
  - Sorption of sulfate on C-S-H
    (*experimental data:* Divet et al. CCR 1998, Barbarulo et al. 2002, Skapa *in progress 2008*
  - Substitution of alumina (Richardson CCR 1994, Chen et al. ICCC 2007, Pardal et al. CCR 2009 & Inorg. Chem. 2012)
- kinetics can be taken into account as function of clinker reactivity over time
  (e.g. Lothenbach et al. CCR 2006 & 2008)

Hydration of OPC with limestone – system creation

Experimental problem part 2:

Blending of the model cement with 5% limestone

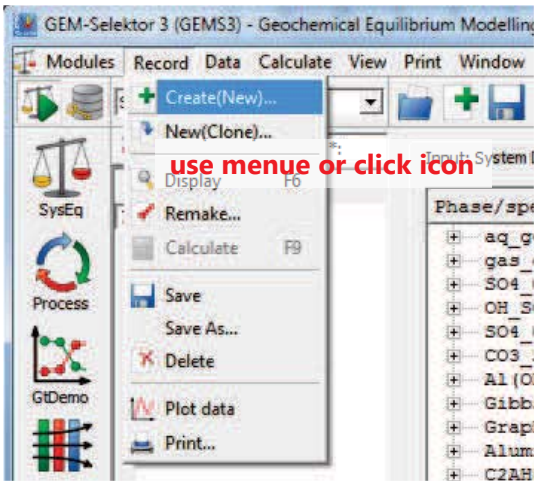
C_3S	81.37 wt.-%		C_3S	77.30 wt.-%
C_2S	1.70 wt.-%		C_2S	1.62 wt.-%
C_3A	11.93 wt.-%	+ 5% $CaCO_3$ →	C_3A	11.33 wt.-%
$CaSO_4$	5.00 wt.-%		$CaSO_4$	4.75 wt.-%
			$CaCO_3$	5.00 wt.-%
w/c	*0.50*			

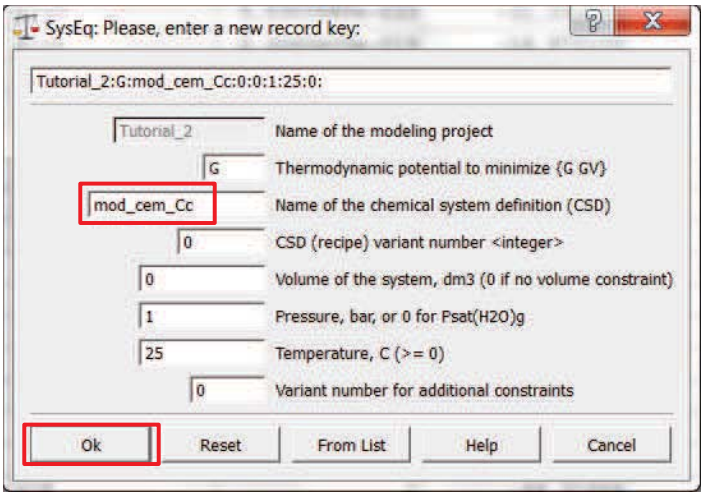
Assumption: 100% hydration

Hydration of OPC with limestone – system creation

Limestone addition:
repeat calculations with 95% OPC blended with 5% $CaCO_3$

Create new system





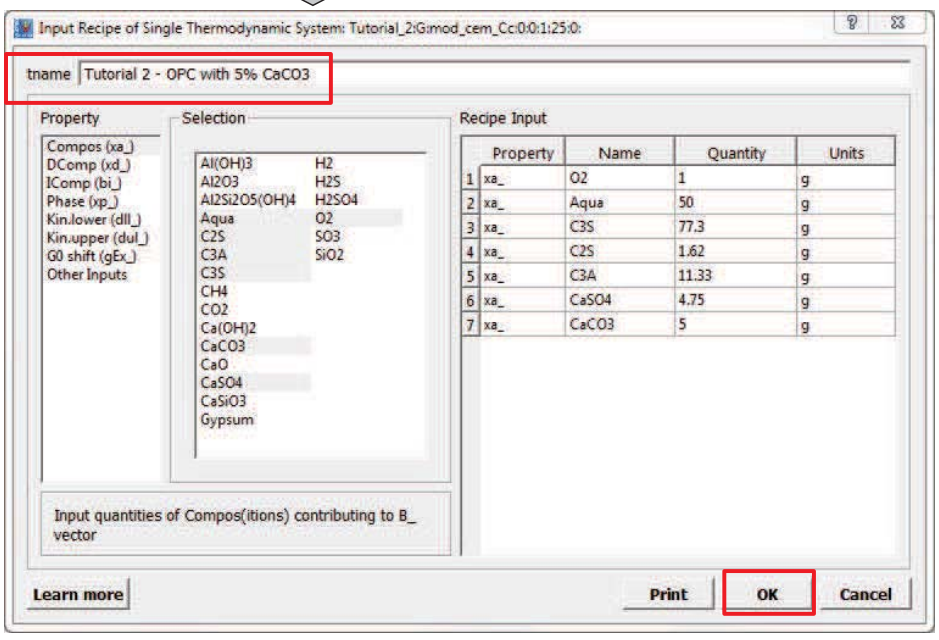
Press OK again to leave the recipe wizard

Hydration of OPC with limestone – system creation

<u>Experimental conditions:</u> React 100 g OPC blended with 5% $CaCO_3$ with 50 g water (add 1 g O_2)

Use recipe wizard

Enter phase composition



Hydration of OPC with limestone - results

Experimental conditions: React 100 g OPC blended with 5% $CaCO_3$ with 50 g water (add 1 g O_2)

Input: System Definition | Results: Equilibrium State

Phase/species	L	T	Amount (mol)	logSI/Activity	Concentration
a aq_gen	37	a	0.57495244	1.582e-10	
g gas_gen	5	g	0.031237771	1.058e-11	
s CSHQ	4	s	0.45695871	1.132e-09	
CSH-JenD		I	0.18665924	0.408482	0.40848163
CSH-JenH		I	0.12383725	0.271003	0.27100314
CSH-TobD		I	0.14033863	0.307114	0.30711446
CSH-TobH		I	0.0061235984	0.0134008	0.01340077
s SO4_OH_AFm	2	s	0	-1	
s OH_SO4_AFm	2	s	0	-1	
s SO4_CO3_AFt	2	s	0.0064282692	-3.818e-11	
tricarboalu		J	0.00061333041	0.0116799	0.095411439
ettringite		M	0.0058149388	0.83947	0.90458856
s CO3_SO4_AFt	2	s	0.0064282692	-3.818e-11	
tricarboalu		M	0.00061333041	0.0116799	0.095411439
ettringite		J	0.0058149388	0.83947	0.90458856
s Al(OH)3mic	1	s	0	-3.024	
s Gibbsite	1	s	0	-2.571	
s Belite	1	s	0	-1.754	
s Aluminate	1	s	0	-38	
s Alite	1	s	0	-13.79	
s C2AH75	1	s	0	-3.99	
s C3AH6	1	s	0	-2.489	
s C4AH13	1	s	0	-3.19	
s C4AH19	1	s	0	-2.741	
s CAH10	1	s	0	-4.99	
s C4AsH12	1	s	0	-1.058	
s C2ASH8	1	s	0	-2.684	
s C4Ac0.5H12	1	s	0	-0.7002	
s C4AcH11	1	s	0.029076338	2.494e-10	
s ettringite	1	s	0	-0.07599	
s Aragonite	1	s	0	-0.1438	
s Calcite	1	s	0.01720015	8.41e-10	
s lime	1	s	0	-9.776	
s Portlandite	1	s	0.46815706	-3.375e-11	
s Anhydrite	1	s	0	-2.97	
s Gypsum	1	s	0	-2.747	

CSH-JenD = $(CaO)_{1.5}\cdot(SiO_2)_{0.6667}\cdot(H_2O)_{2.5}$
CSH-JenH = $(CaO)_{1.333}\cdot(SiO_2)_1\cdot(H_2O)_{2.1667}$
CSH-TobD = $[(CaO)_{1.25}\cdot(SiO_2)_1\cdot(H_2O)_{2.75}]_{0.6667}$
CSH-TobH = $(CaO)_{0.6667}\cdot(SiO_2)_1\cdot(H_2O)_{1.5}$

C-S-H solid solution
1.63 CaO · SiO_2 · 2.88 H_2O

AFt solid solution:
0.905 mol SO_4-ettringite
0.095 mol CO_3-ettringite
=> $Ca_6Al_2(OH)_{12}(SO_4)_{2.715}(CO_3)_{0.285}\cdot 26H_2O$

Monocarboaluminate (CO_3-AFm)

=> Significant mineralogical changes due to limestone addition

Hydration of OPC with limestone - results

Experimental conditions: React 100 g OPC blended with 5% $CaCO_3$ with 50 g water (add 1 g O_2)

Predicted phase assemblage *(hydrated cement paste)*:

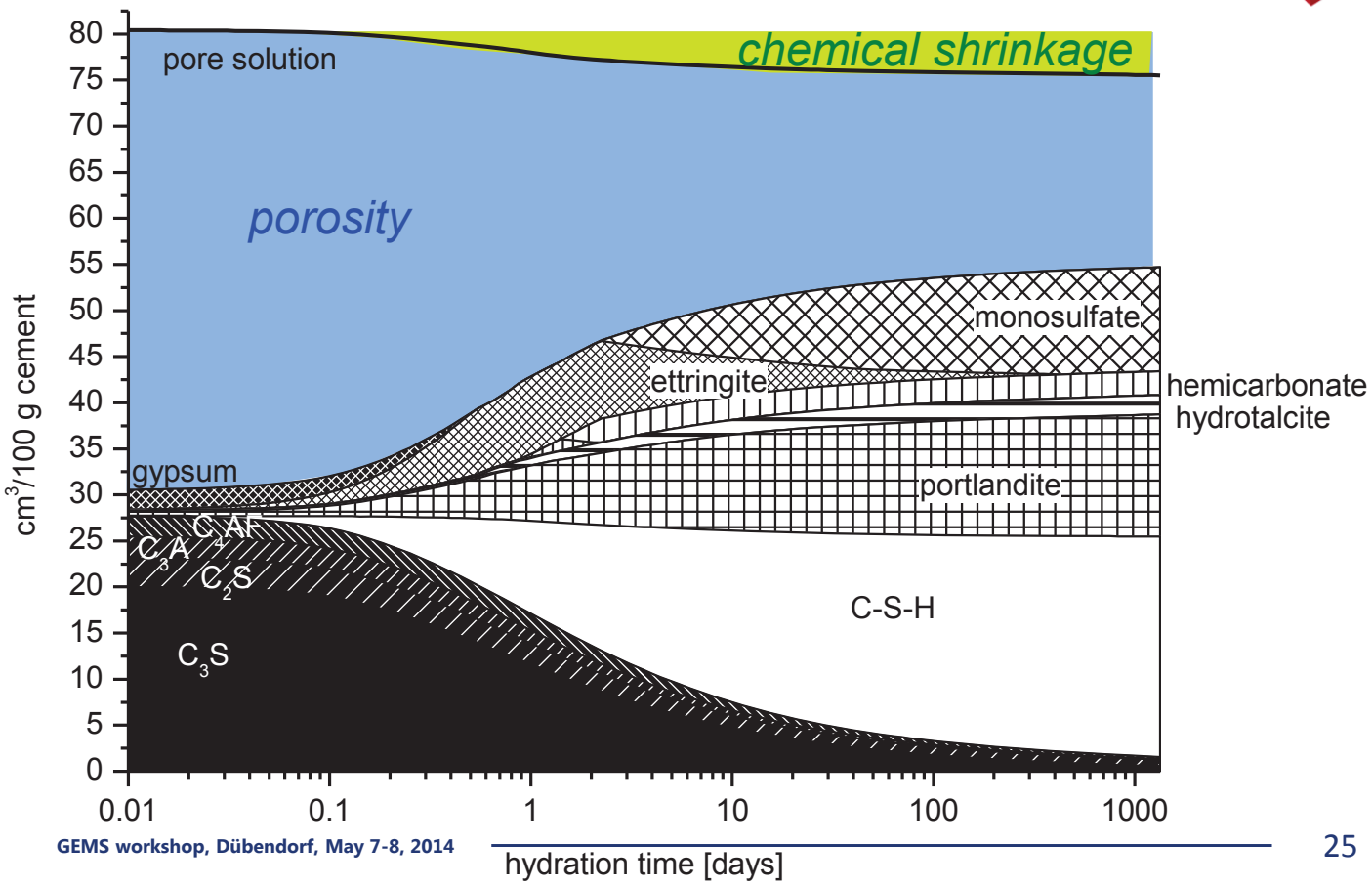
- C-S-H (solid solution)
- SO_4-AFt (solid solution)
- CO_3-AFm
- Calcite
- Portlandite

Tutorial 2 - OPC with 5% CaCO3

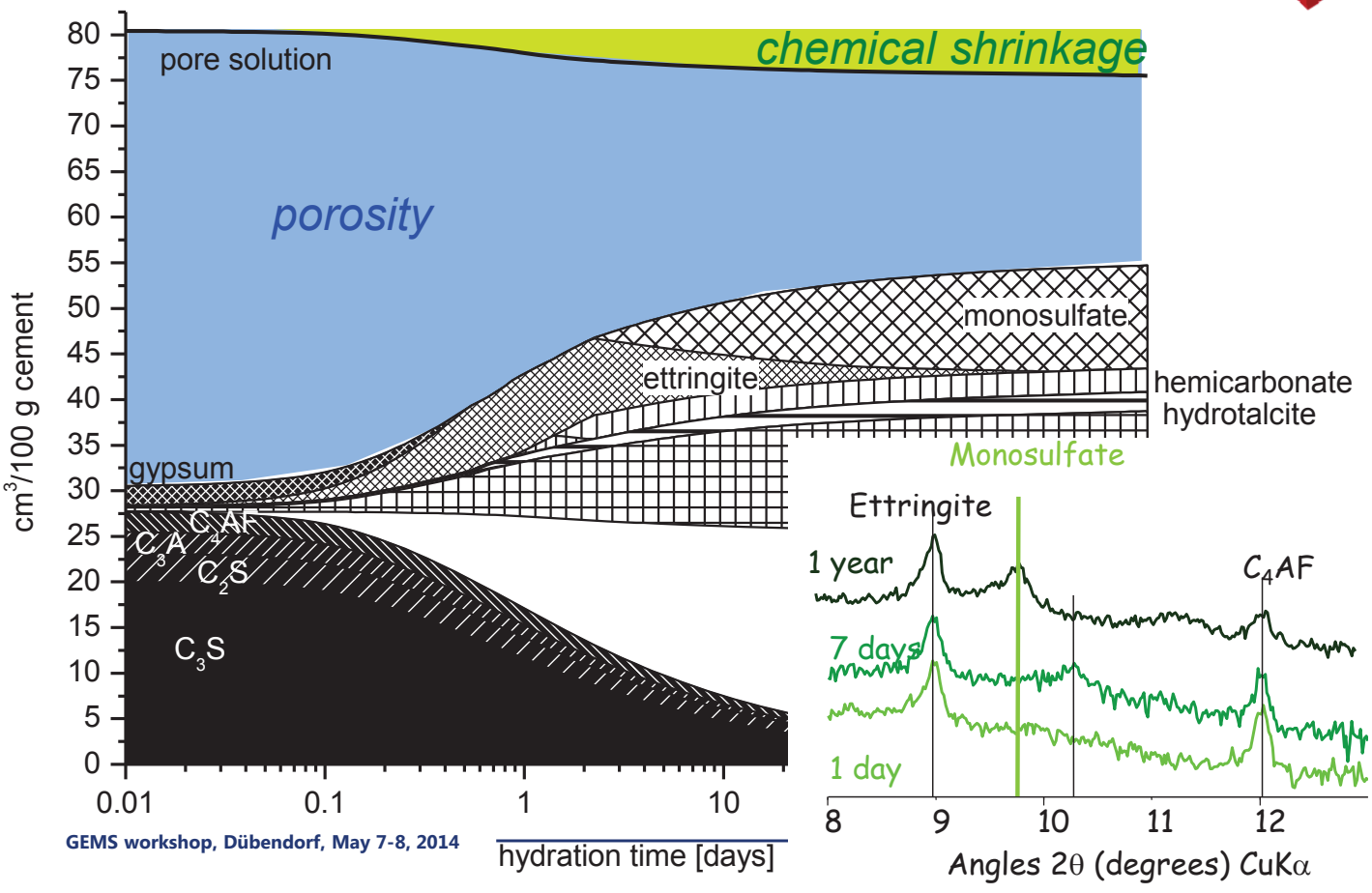
	PHnam	L1	Xa	Fa	hVol	phM
0	a aq_gen	37	0.57495244	1.5816157e-010	10.373392	10.363212
1	g gas_gen			022e-011	774.37532	0.99957118
2	s CSHQ				31.476055	70.696579
3	s SO4_OH_AFm	2	0	-1	0	0
4	s OH_SO4_AFm	2		-1	0	0
5	s SO4_CO3_AFt	2		011	4.5102464	8.0018486
6	s CO3_SO4_AFt	2	0.0064282692	-3.8178544e-011	4.5102464	8.0018486
7	s Al(OH)3mic	1	0	-3.0243936	0	0
8	s Gibbsite	1	0	-2.5712435	0	0
9	s Belite	1	0	-1.7543913		
10	s Aluminate	1	0	-38.001866		
13	s C3AH6	1	0	-2.4894055	0	0
14	s C4AH13	1	0	-3.1903608		
15	s C4AH19	1	0	-2.7411143		
16	s CAH10	1	0	-4.9897519	0	0
17	s C4AsH12	1	0	-1.0579398	0	0
18	s C2ASH8	1			0	0
19	s C4Ac0.5H12	1			0	0
20	s C4AcH11	1	0.029076338	2.4943526e-010	7.6167795	
21	s ettringite			07599459	0	
22	s Aragonite			3216	0	
23	s Calcite	1	0.01720015	8.4095527e-010	0.63527032	1.7215114
24	s lime	1	0	-9.7760819	0	0
25	s Portlandite	1	0.46815706	-3.3746833e-011	15.477272	34.68702

$V_{pore\ sol.}$ = 10.37 cm³
$m_{pore\ sol.}$ = 10.36 g
$V_{C\text{-}S\text{-}H}$ = 31.48 cm³
$m_{C\text{-}S\text{-}H}$ = 70.70 g
$V_{AFt(ss)}$ = 9.02 cm³
$m_{AFt(ss)}$ = 16.00 g
$M_{calcite}$ = 1.72 g => 5.00-1.72 = **3.28 g** calcite had reacted
$m_{CO3\text{-}AFm}$ = 16.53g
$V_{CO3\text{-}AFm.}$ = 7.62 cm³
$V_{calcite}$ = 0.64 cm³
$m_{calcite}$ = 1.72 g
$V_{Portlandite}$ = 15.48 cm³
$m_{portlandite}$ = 34.69 g

Comparison experimental data and modelling: Hydration of OPC without limestone

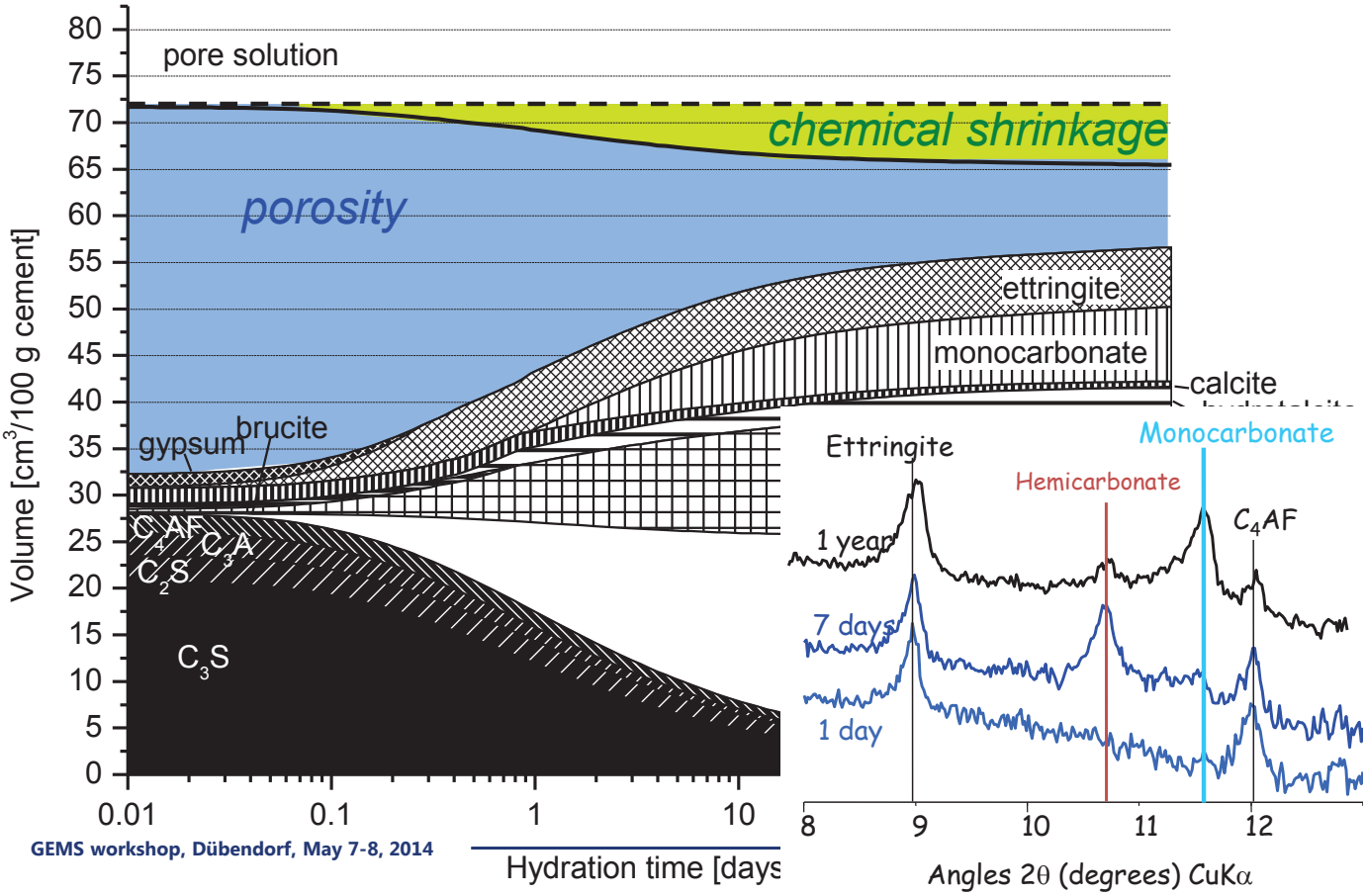


Comparison experimental data and modelling: Hydration of OPC without limestone



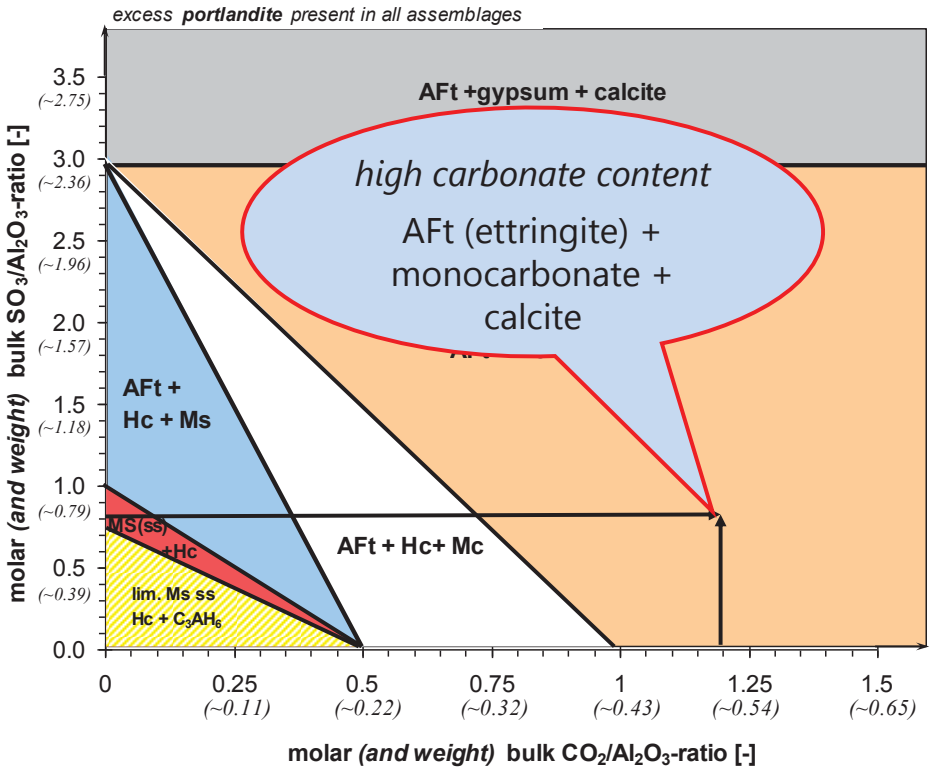
GEMS workshop, Dübendorf, May 7-8, 2014

Comparison experimental data and modelling: Hydration of OPC with limestone



Hydration of OPC with limestone – phase diagram

Estimation of phase assemblages



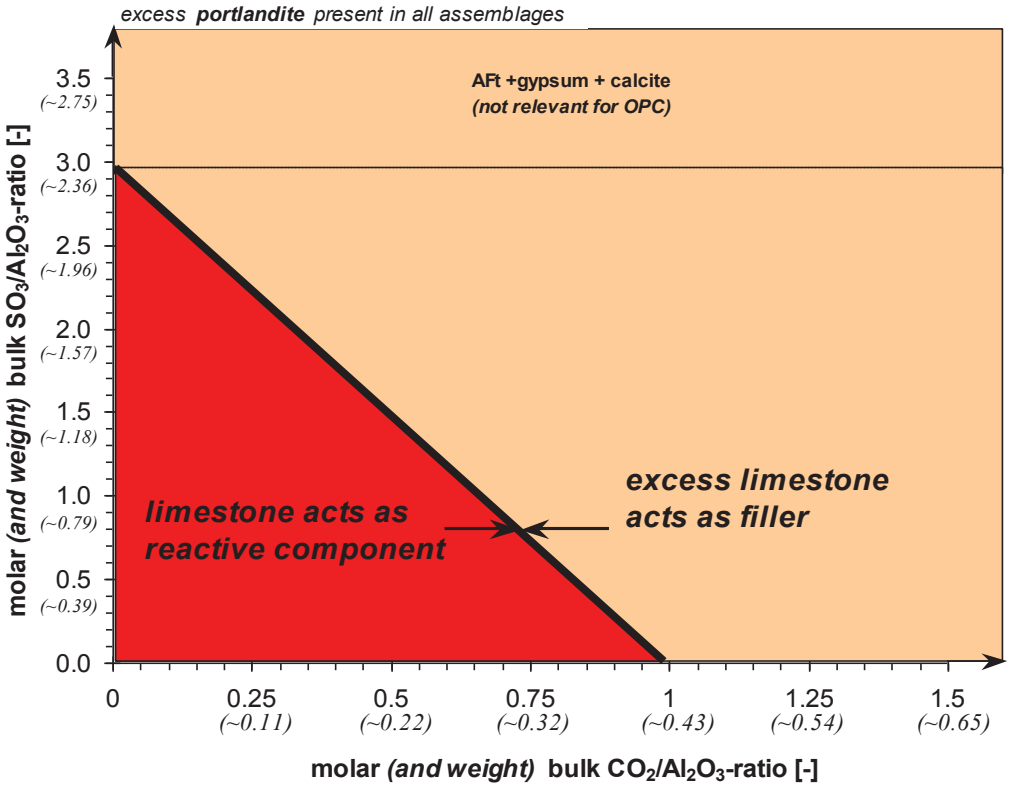
Our cement:
molar SO_3/Al_2O_3=0.82
weight SO_3/Al_2O_3=0.64

molar CO_2/Al_2O_3= 1.19
weight CO_2/Al_2O_3= 0.52

=> Formation of monocarboaluminate, AFt and calcite expected

Hydration of OPC with limestone – phase diagram

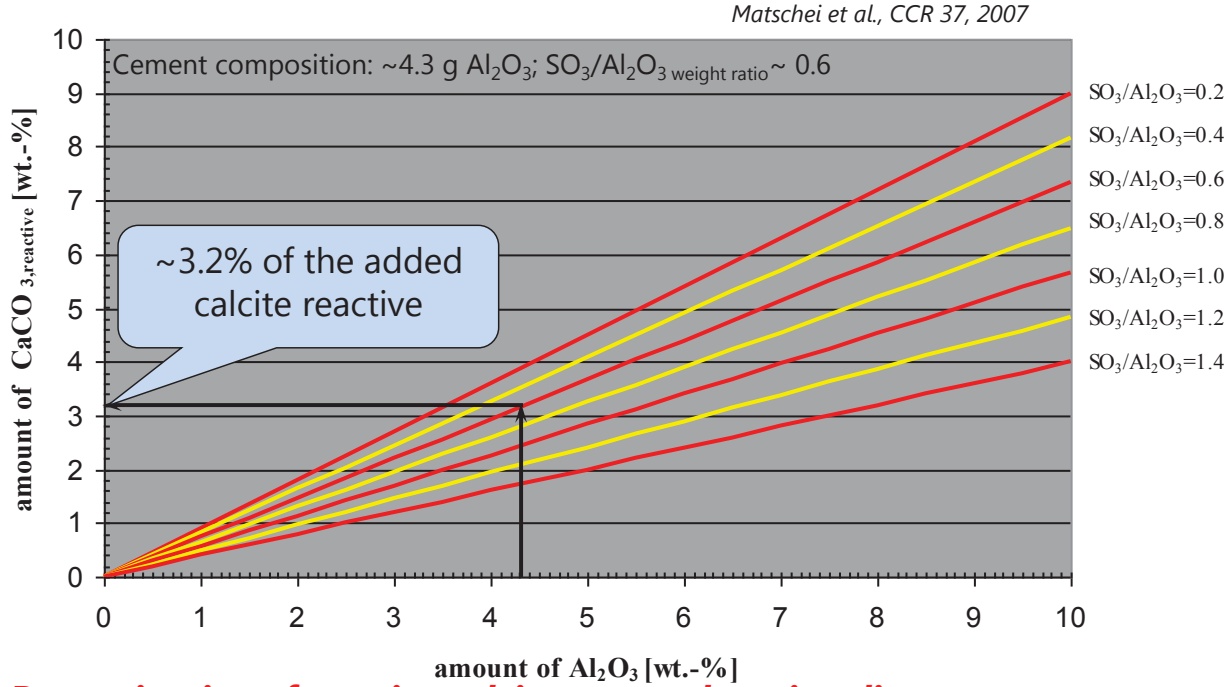
Distinction between limestone as reactive admixture and as filler



Hydration of OPC with limestone – phase diagram

Previous calculations $M_{calcite}$ = 1.81 g => 5-1.71 = 3.19g calcite have reacted

Matschei et al., CCR 37, 2007



=> *Determination of reactive calcite content by using diagrams based on thermodynamic phase predictions*

Appendix – siliceous hydrogarnet

Predicted stable phase assemblage

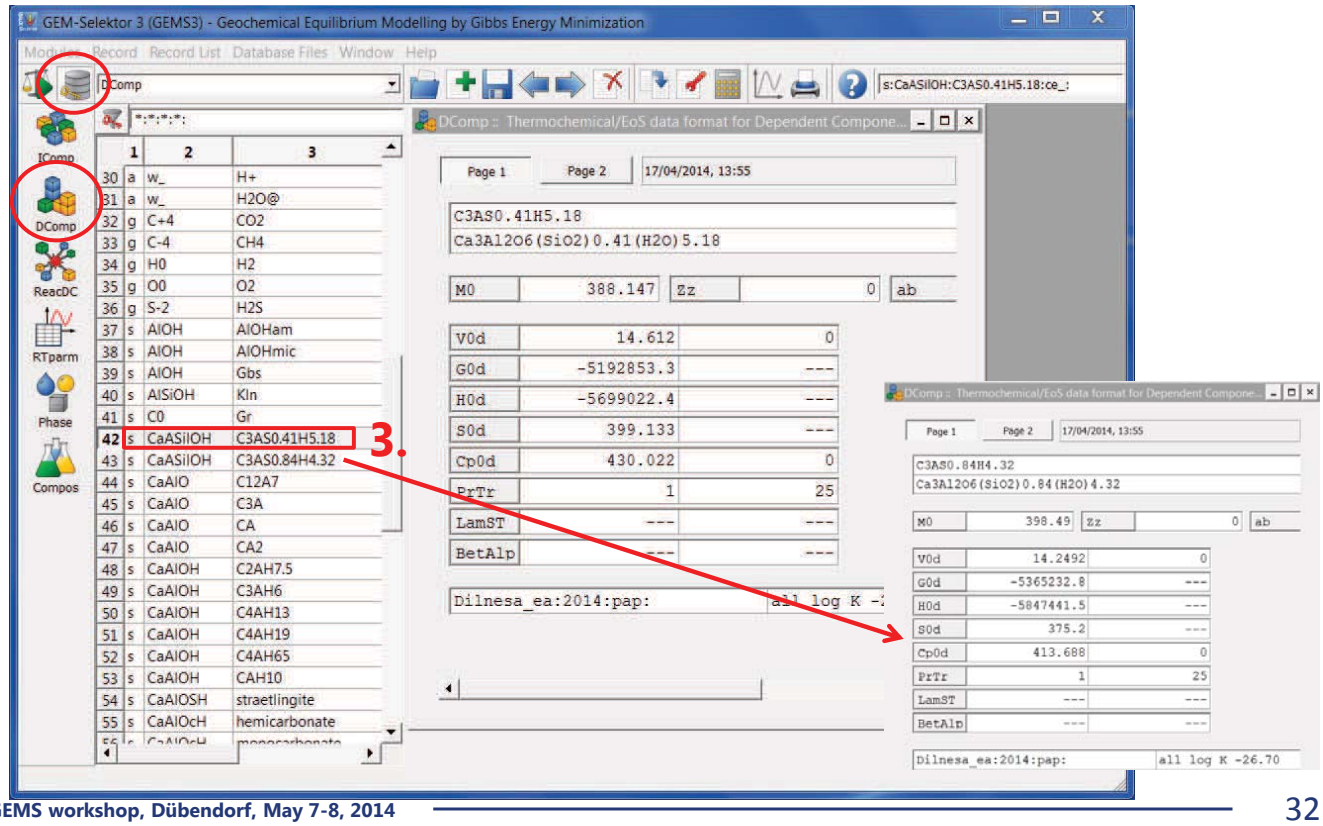
Possibility of formation of siliceous hydrogarnet in the system $CaO\text{-}SiO_2\text{-}Al_2O_3\text{-}CaSO_4\text{-}CaCO_3\text{-}H_2O$ at 25°C

- according to the literature the formation of siliceous hydrogarnet is likely in heat treated concretes
- AFm phases are abundant and generally observed in cement paste cured at 25°C
- knowledge on thermodynamic properties of solid solutions between different hydrogarnets containing Fe, Ca, Si has improved recently (Dilnesa et. al. CCR 2014a+b)
- formation of siliceous hydrogarnet apparently kinetically restraint

To reproduce reality, the formation of siliceous hydrogarnet is by default supressed in GEMS. The data however is included in the cement database (DComp) and can be introduced (Phase) if desired.

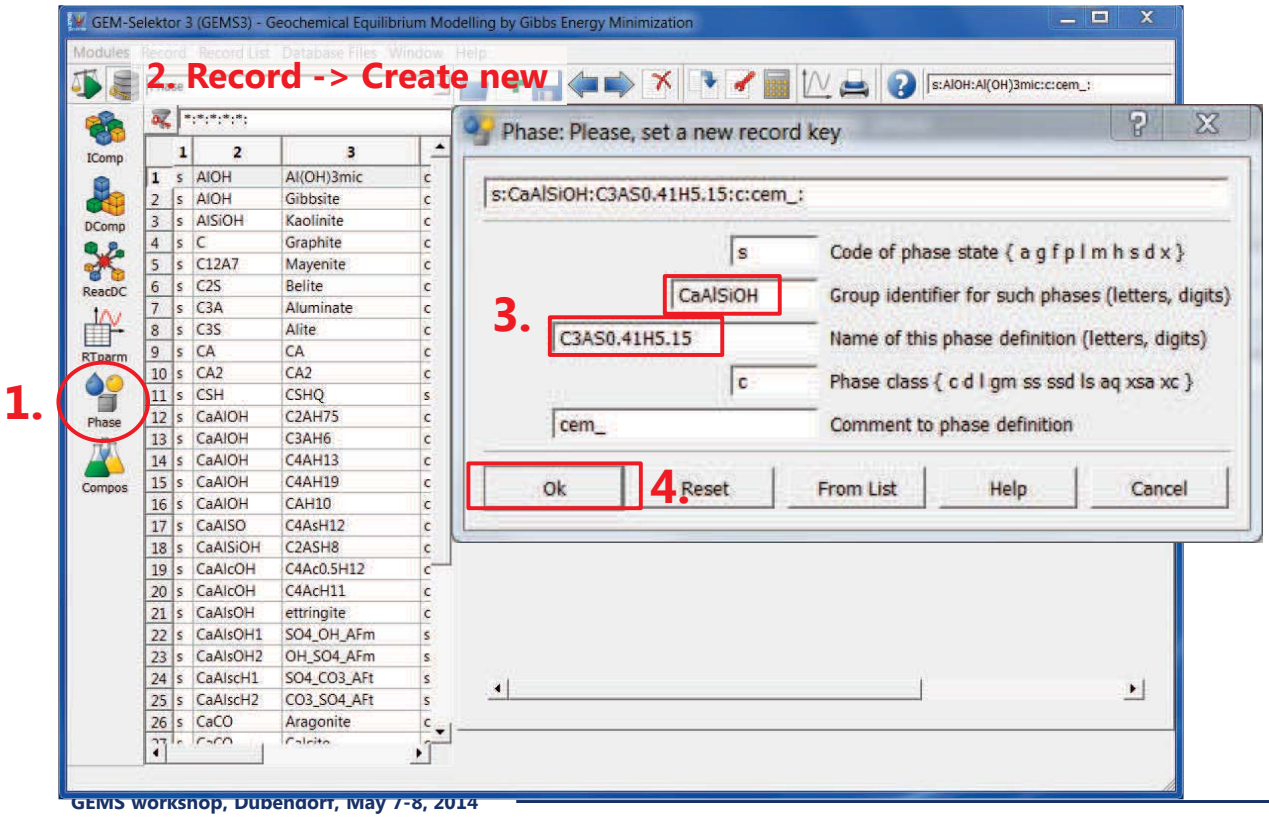
Appendix – siliceous hydrogarnet

Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



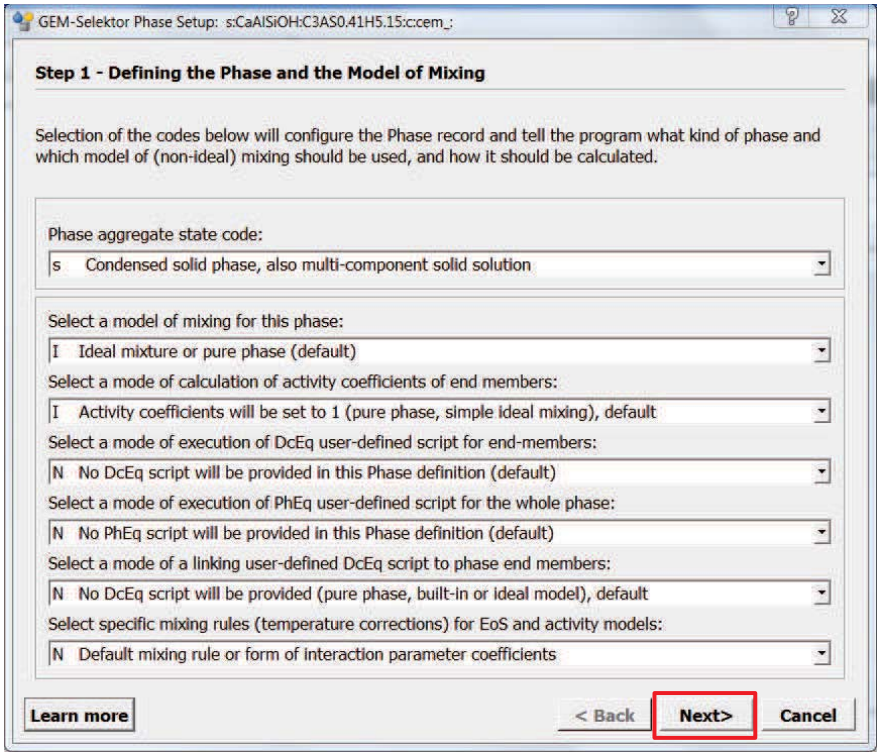
Appendix – siliceous hydrogarnet

Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



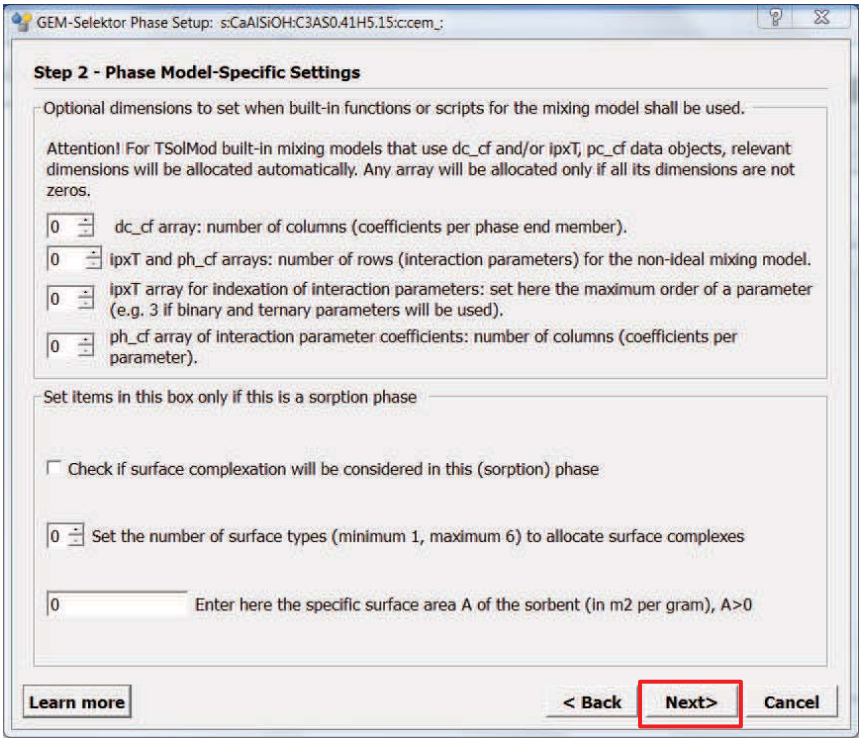
Appendix – siliceous hydrogarnet

Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



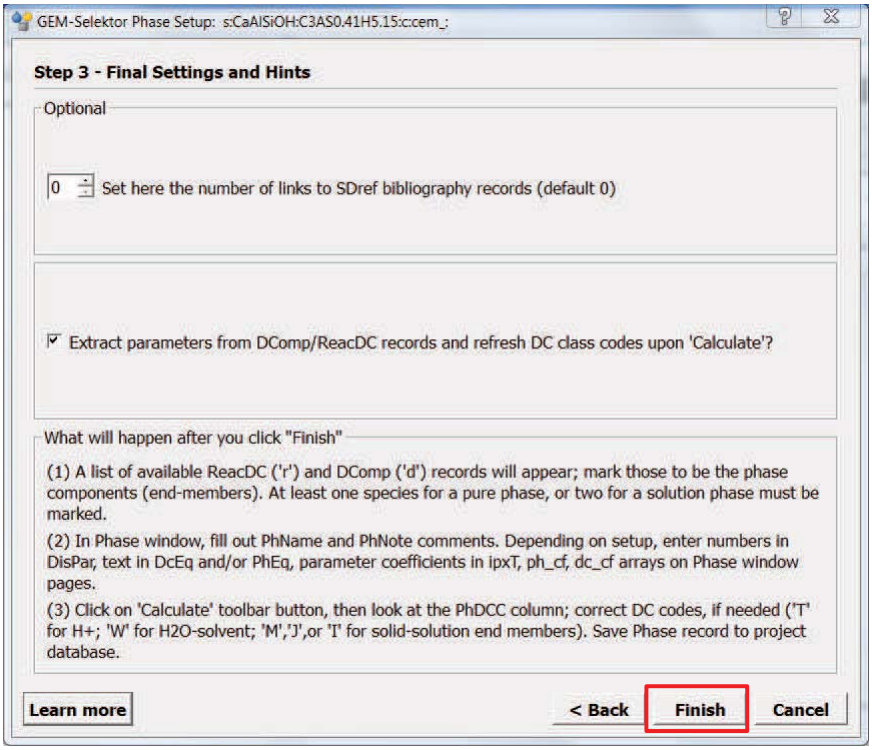
Appendix – siliceous hydrogarnet

Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



Appendix – siliceous hydrogarnet

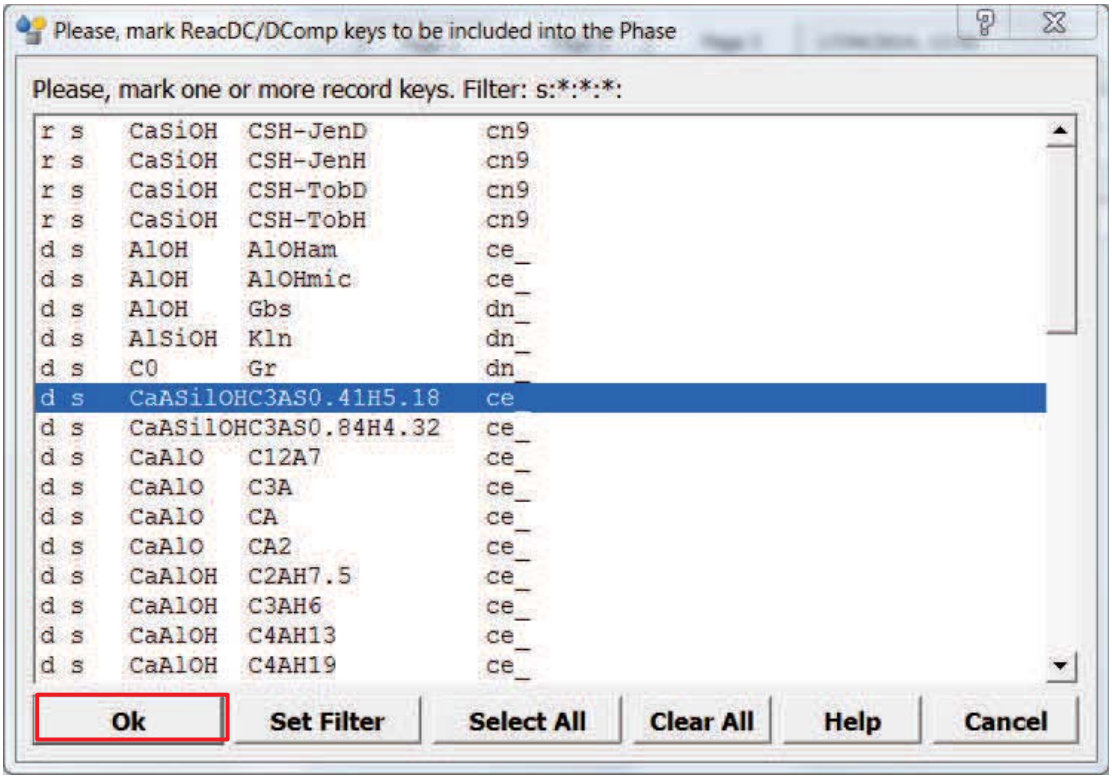
Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



Appendix – siliceous hydrogarnet

EMPA

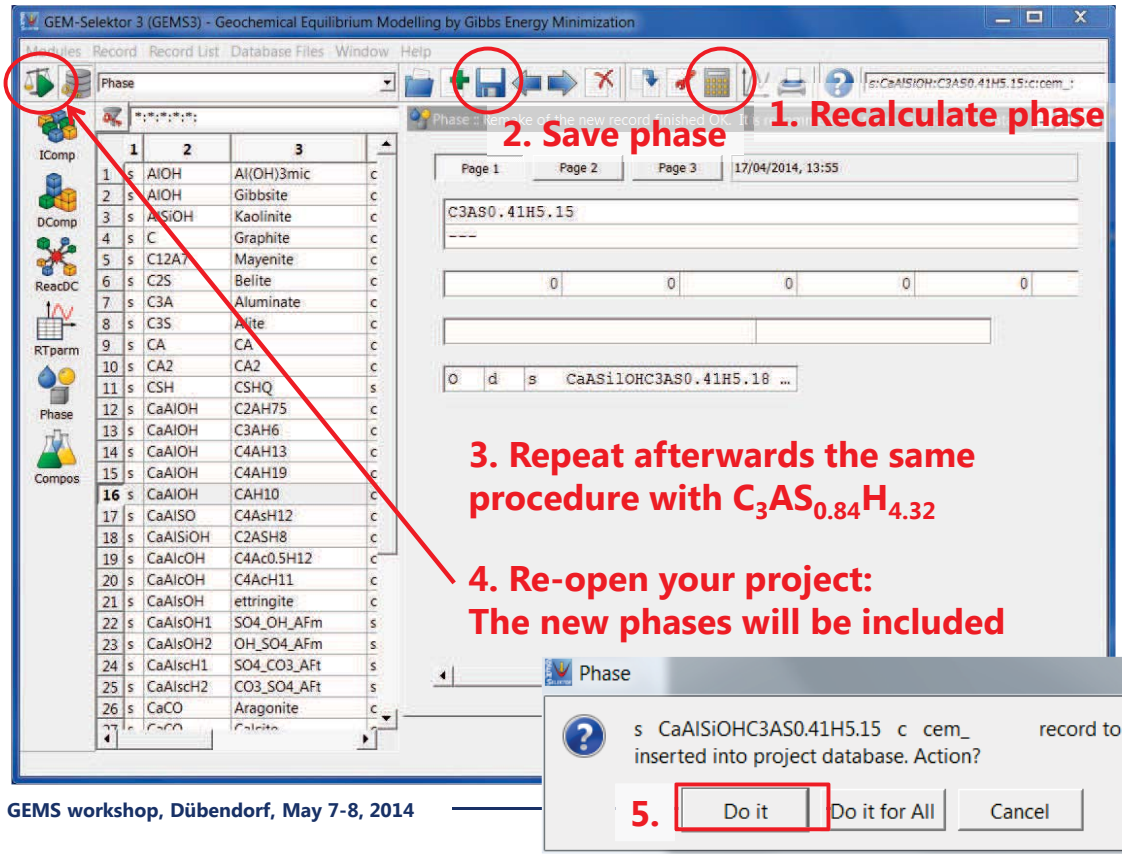
Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



Appendix – siliceous hydrogarnet

EMPA

Import of siliceous Al-hydrogarnets (Dcomp -> Phase)



Appendix – siliceous hydrogarnet

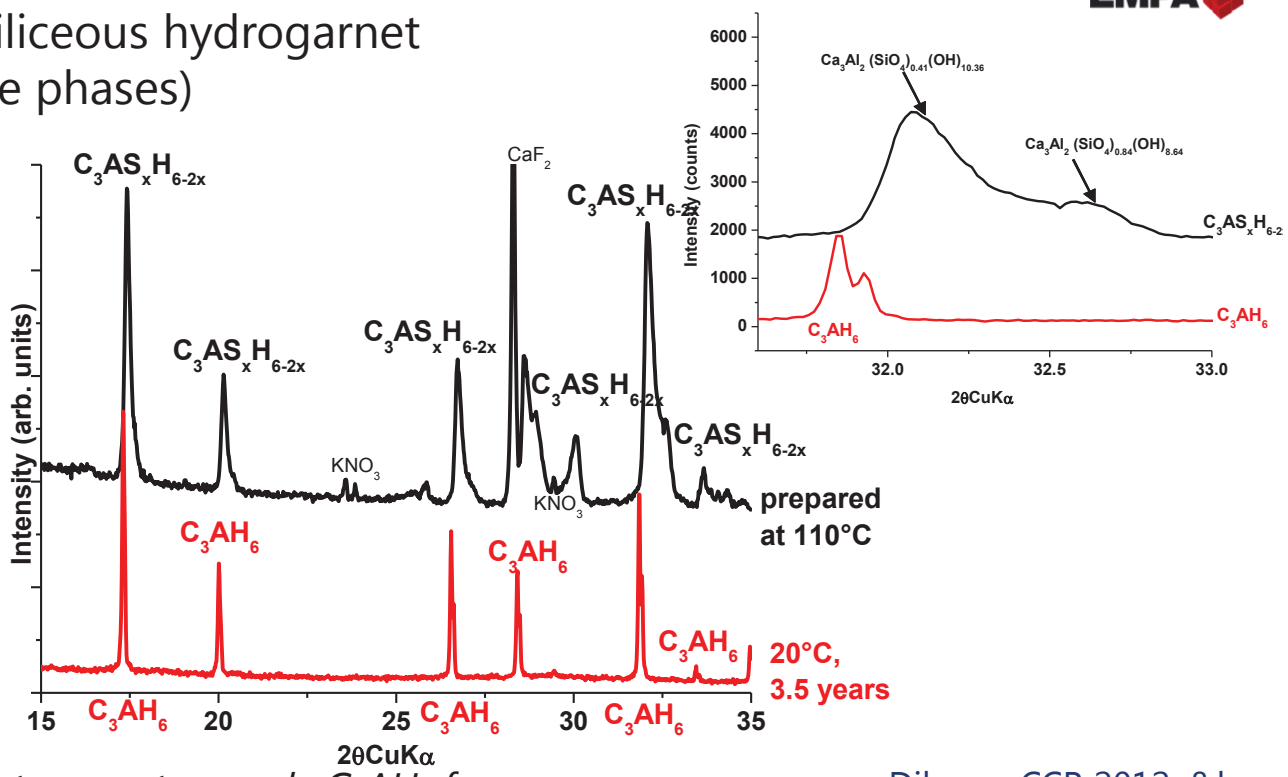
Predicted stable phase assemblage in presence of Si-Hg (for OPC+calcite)

Input: System Definition | Results: Equilibrium State

Phase/species	L	T	Amount (mol)
a aq_gen	37	a	0.77375998
g gas_gen	5	g	0.031233137
s CSHQ	4	s	0.44101851
s SO4_OH_AFm	2	s	0
s OH_SO4_AFm	2	s	0
s SO4_CO3_AFt	2	s	0.0061637368
s CO3_SO4_AFt	2	s	0.0061637368
s Al(OH)3mic	1	s	0
s Gibbsite	1	s	0
s Belite	1	s	0
s Aluminate	1	s	0
s Alite	1	s	0
s C2AH75	1	s	0
s C3AH6	1	s	0
s C4AH13	1	s	0
s C4AH19	1	s	0
s CAH10	1	s	0
s C4AsH12	1	s	0
s C2ASH8	1	s	0
s C3AS0.41H5.15	1	s	0.029605411
s C3AS0.84H4.32	1	s	0
s C4Ac0.5H12	1	s	0
s C4AcH11	1	s	0
s ettringite	1	s	0
s Aragonite	1	s	0
s Calcite	1	s	0.047863404
s lime	1	s	0
s Portlandite	1	s	0.48783282
s Anhydrite	1	s	0
s Gypsum	1	s	0

Appendix – siliceous hydrogarnet

Al-siliceous hydrogarnet (pure phases)



Room temperature: only C_3AH_6 forms;
$C_3AS_xH_{6-2x}$ forms at 110 C°, but also stable at 20 °C

Dilnesa, CCR 2012a&b

Appendix – siliceous hydrogarnet

Al-siliceous hydrogarnet (OPC blended with fly ash)

Deschner, CCR 2013