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## Design of a hybrid leaching process for mineral carbonation of magnesium silicates: learnings and issues raised from combined experimental and geochemical modelling approaches

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# Design of a hybrid leaching process for mineral carbonation of magnesium silicates: learnings and issues raised from combined experimental and geochemical modelling approaches

Carine Julcour<sup>1</sup>, Florent Bourgeois<sup>1</sup>, Laurent Cassayre<sup>1</sup>, Julien Leclaire<sup>2</sup>, Solène Touzé<sup>3</sup>, Martin Cyr<sup>4</sup>, France Bailly<sup>5</sup>

<sup>1</sup>LGC, Toulouse, France ; <sup>2</sup>ICBMS, Villeurbanne, France ; <sup>3</sup>BRGM, Orléans, France ; <sup>4</sup>LMDC, Toulouse, France ; <sup>5</sup>CNRT, Nouméa, France



**CO<sub>2</sub> CARMEX**  
 French National Research Agency  
 ANR (ANR CO<sub>2</sub> 2009-2012)

*Worldwide potential of MC (GIS)  
 Examination of MC mechanisms  
 Proof of concept of selected MC route  
 Environmental assessment (LCA)*



**New-Caledonia as prime candidate for MC deployment**



**CARBOSCORIES I**  
 National Centre for Technology Research  
 CNRT "Nickel and its environment"  
 (2015-2016)

*Potential of Ni slags for MC hybrid process  
 Balance analysis for two metallurgy plants*



**CARBOSCORIES II**  
 Caledonian Energy Agency  
 (2018-2021)



*Bench-scale continuous pilot  
 Valorisation route for MC products*

**CO<sub>2</sub>EMR**  
 Collaborative work

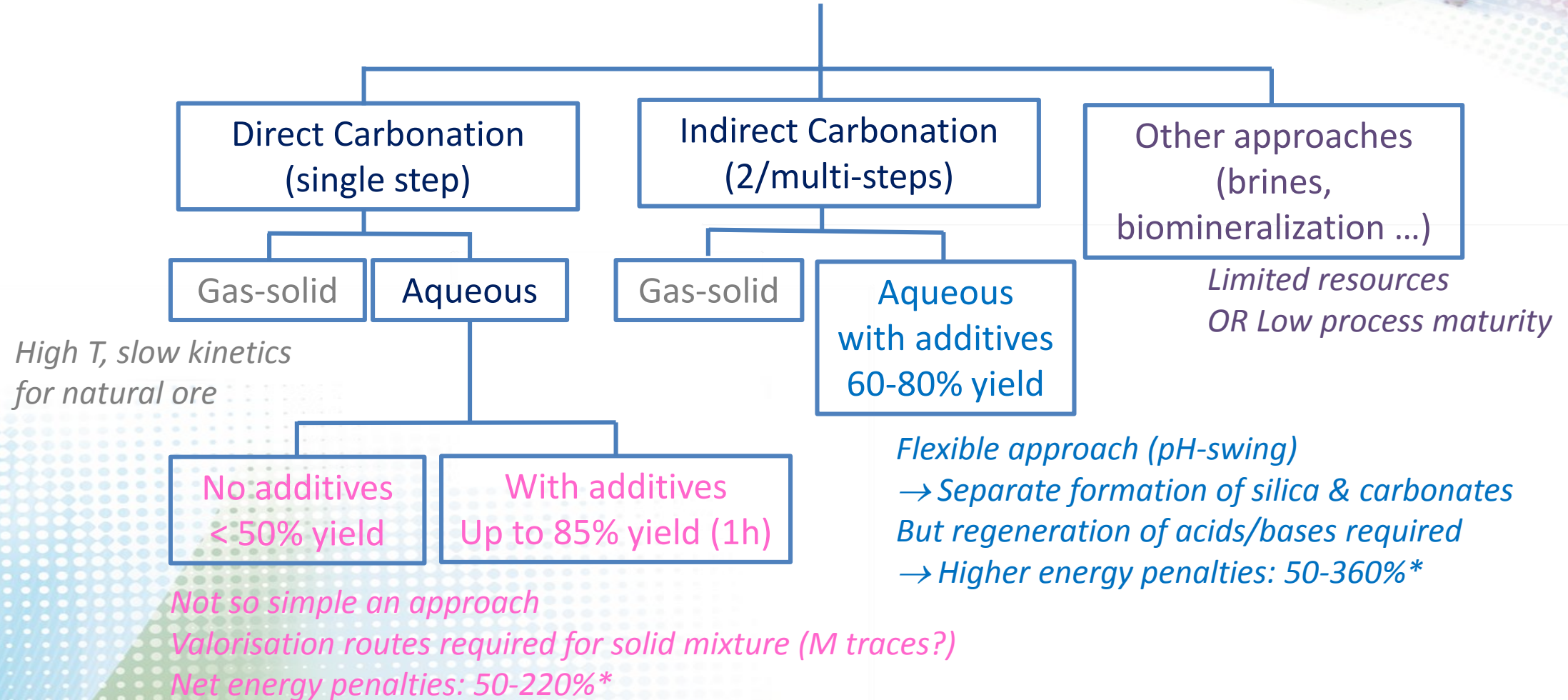


*CO<sub>2</sub> Enhanced Metal Recovery*





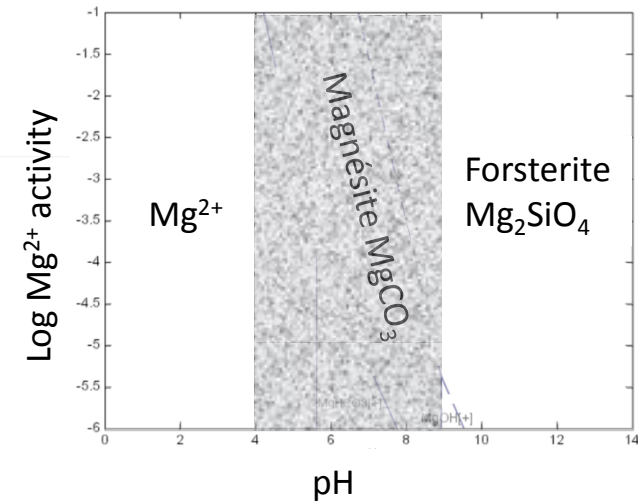
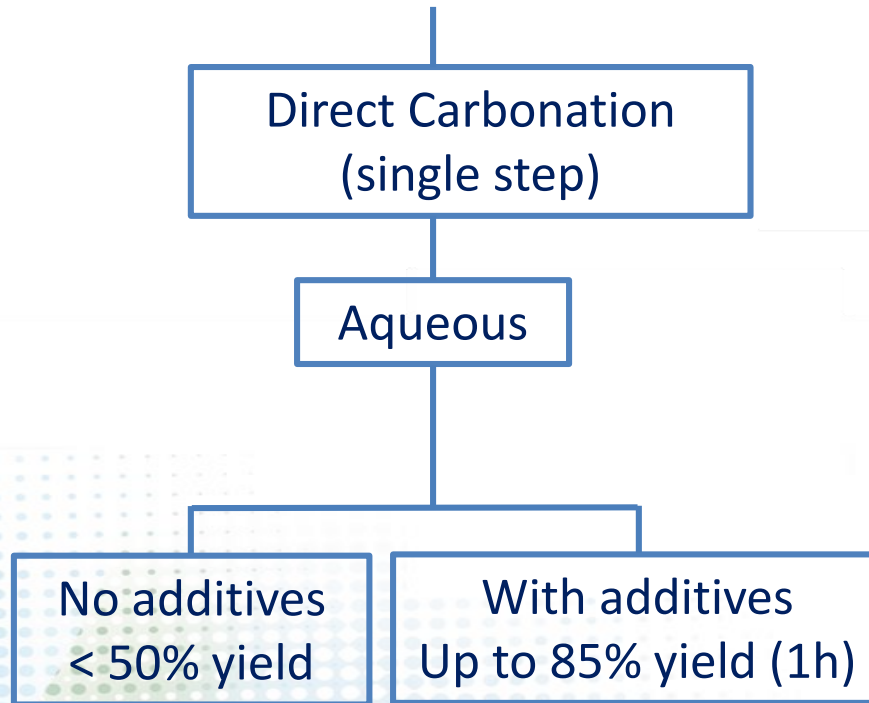
## Ex-situ Mineral Carbonation pathways



Adapted from *Sanna et al. (2014)*

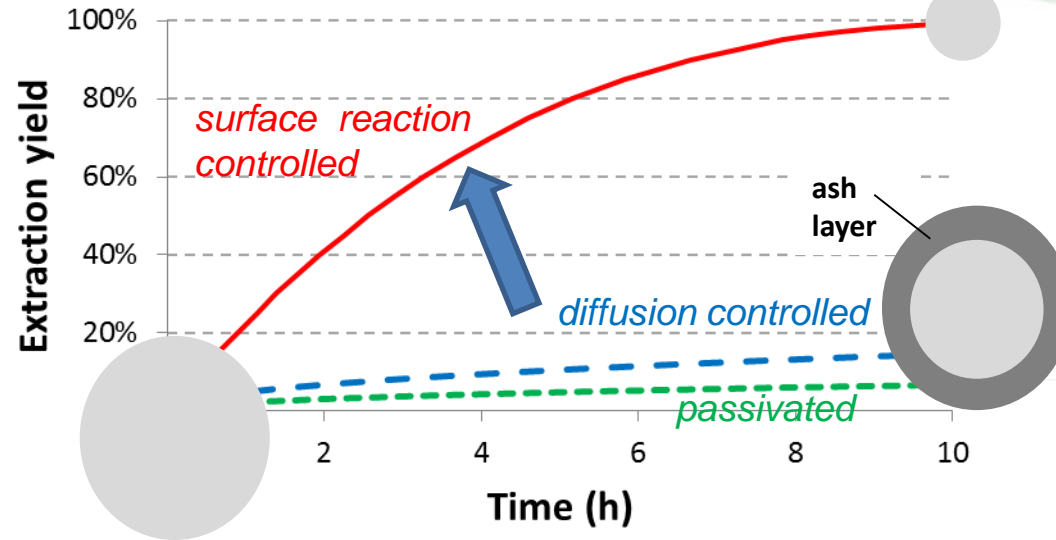
\*based on < 2010 literature routes for 154 MWe coal-fired power plant producing 1Mt-CO<sub>2</sub>/yr

## Studied MC pathways



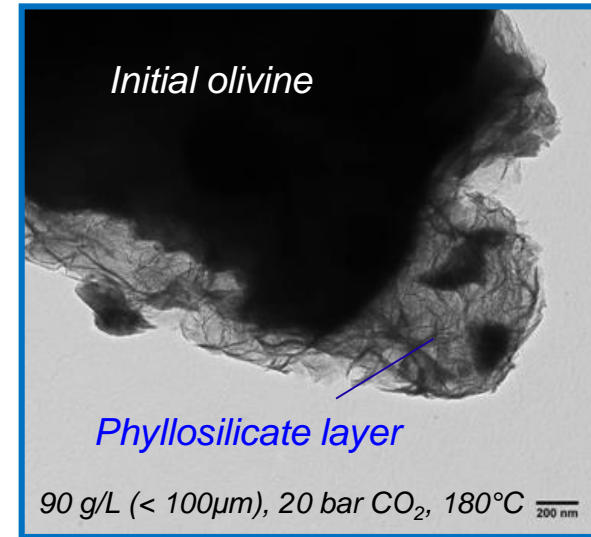
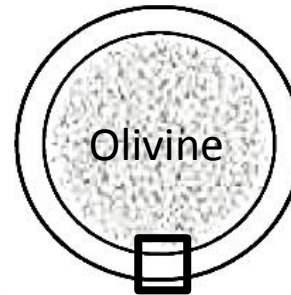
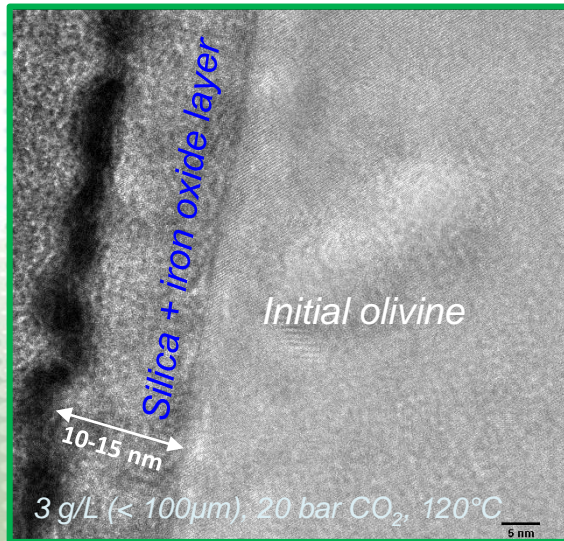


# Surface leach layer & mechanisms



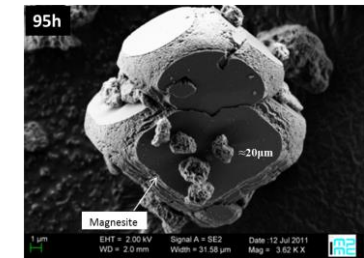
Passivating

→ No carbonate



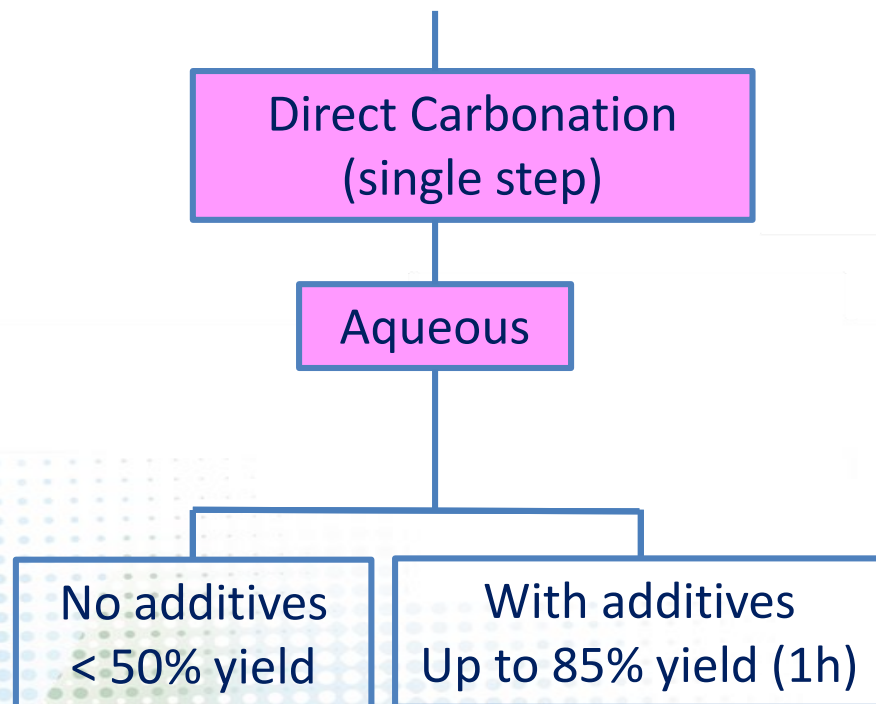
Diffusion controlling

Carbonation yield < 10% (90 g/L, 95 h)



FIB cross-sections of olivine particle after leaching (TEM) (Bodenan et al., 2014)

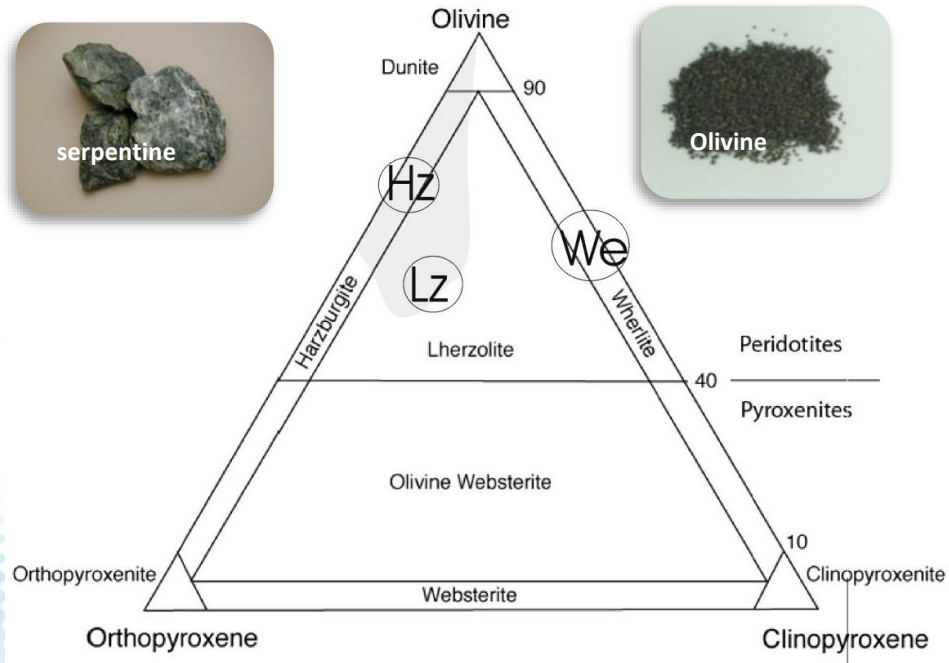
## Studied MC pathways



# EXPERIMENTAL METHODOLOGY



## Ores



**Bodenan et al. (2014)**



### Serpentinisation degree up to 90%

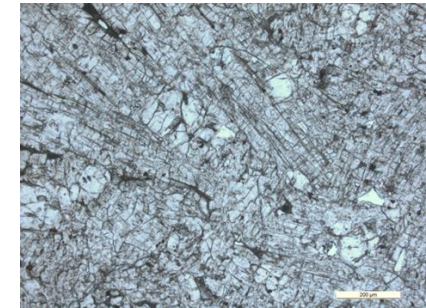
Name	Origin	Dominating phases
Harzburgite (Hz1)	New Caledonia	Serpentine (~ 90%) >> olivine > orthopyroxene
Harzburgite (Hz2)	New Caledonia	Serpentine (~ 90%) >> olivine > orthopyroxene
Wherlite (We)	New Caledonia	Serpentine (~ 50%) > olivine > clinopyroxene
Lherzolite (Lz)	France (Pyrenees)	Serpentine (~ 50%) > olivine > clino/ortho-pyroxene
Olivine	Austria (Magnolithe)	Synthetic olivine (from high T dunite processing)

30.8 < MgO < 47.4%, 39.3 < SiO<sub>2</sub> < 46.3%, 7.3 < Fe<sub>2</sub>O<sub>3tot</sub> < 9.8%,  
0.2 < CaO < 5.0%, 0.2 < Al<sub>2</sub>O<sub>3</sub> < 4.0%

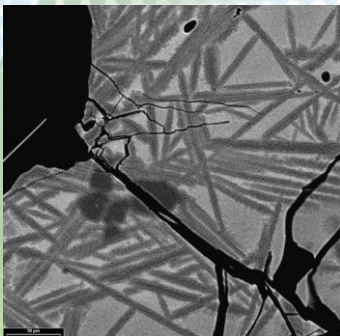
## Slags



From Ni pyrometallurgy plant (Koniambo)  
KNS slowly cooled under ambient conditions, SLN quenched by seawater



Name	Origin	Dominating phases
KNS	New Caledonia	Pyroxene: proto-enstatite, clinoenstatite, forsterite
SLN	New Caledonia	Vitreous fraction >> olivine



MgO ~ 30%, SiO<sub>2</sub> ~ 52%, Fe<sub>2</sub>O<sub>3tot</sub> ~ 13%,  
CaO < 0.5%, Al<sub>2</sub>O<sub>3</sub> ~ 2%

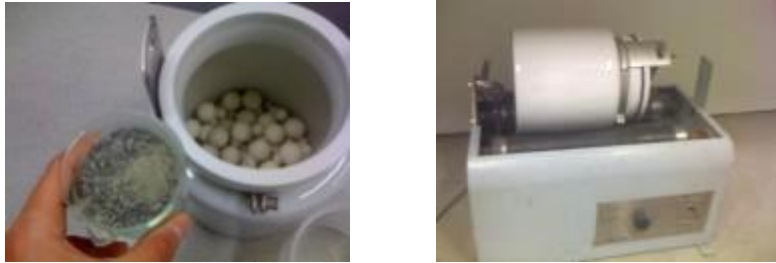


# Solid preparation

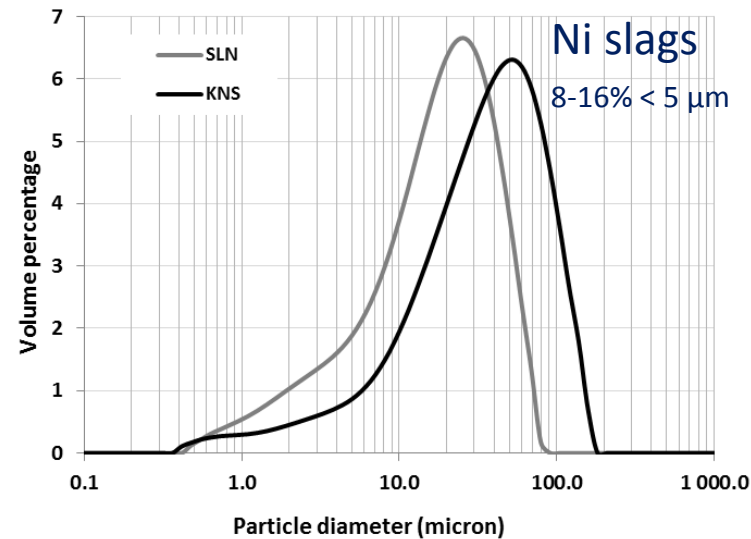
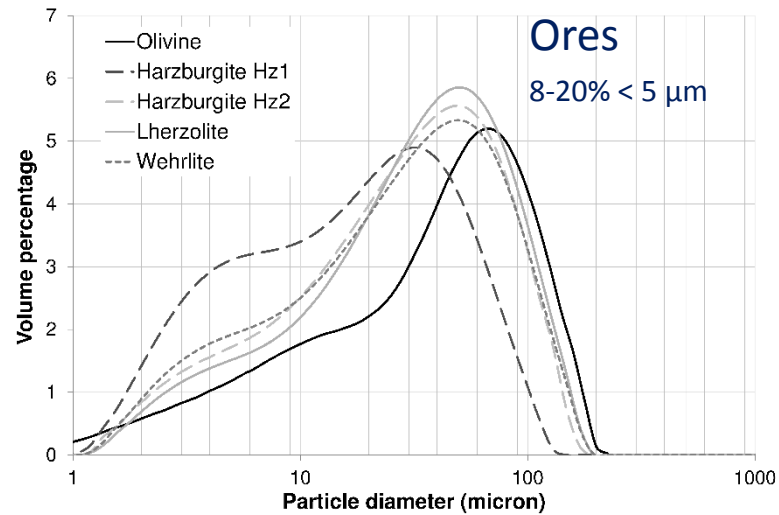
➤ Mechanical sampling



➤ Grinding (4h in ball mill) & sieving < 100 μm



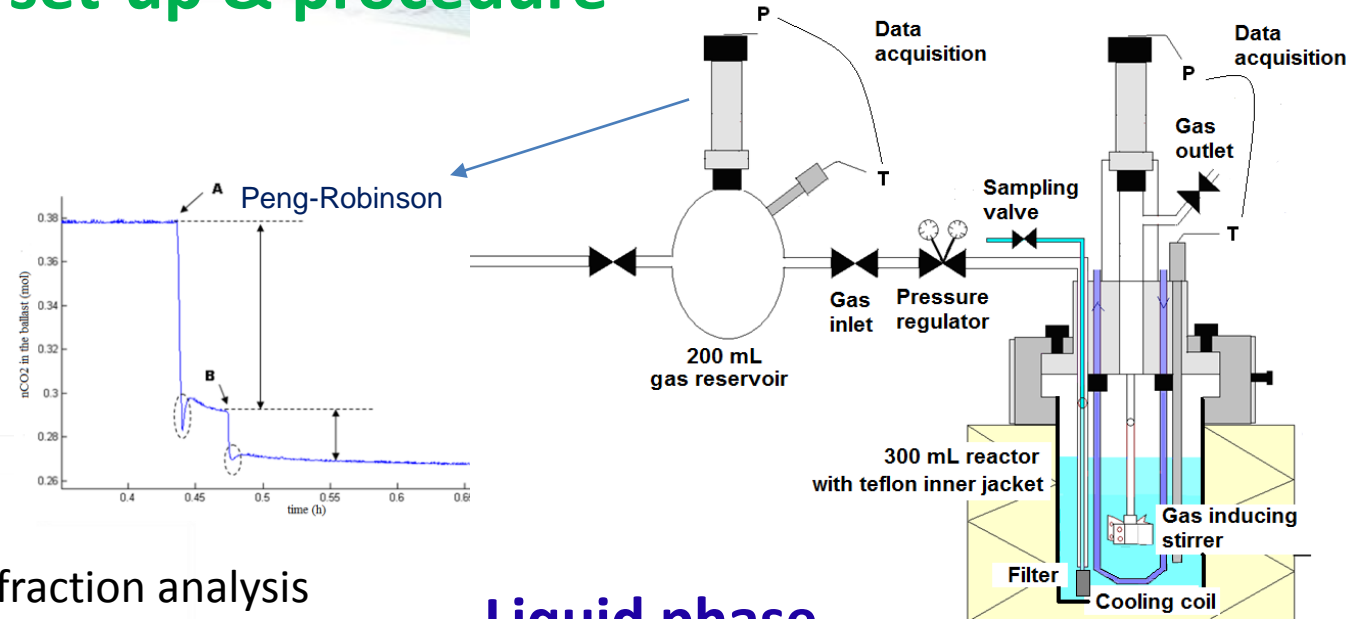
➤ Initial particle size distribution



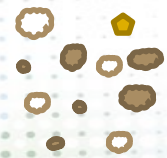


# Carbonation set-up & procedure

- 300 mL autoclave reactor
- Controlled atmosphere ( $P_{CO_2}$ ) and T
- Gas auto-dispersing stirrer (800 rpm)
- Continuous recording of  $CO_2$  consumption (24 h)

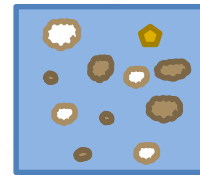


## Solid phase



drying (70°C)

Laser diffraction analysis  
Filtration (0.2 μm)



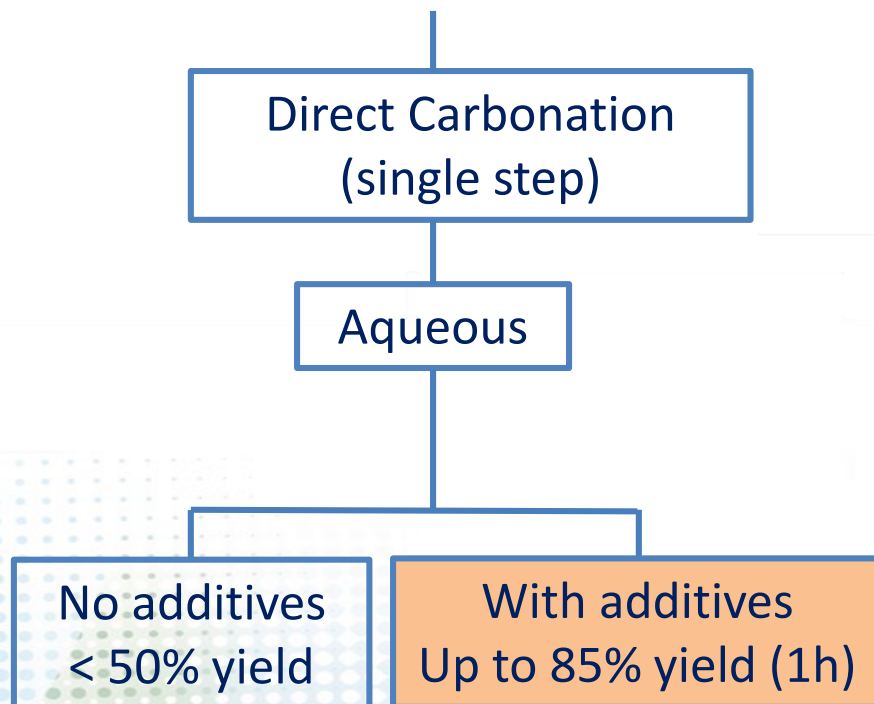
## Liquid phase



- Identification of solids formed: SEM(TEM)/EDX and elemental mapping (Mg, Si, O), X-Ray Diffraction, Raman and IR (DRIFT) Spectroscopy, ICP/AES (after acid dissolution)
- Quantification of carbonated products: elemental analysis (C content), ThermoGravimetry Analysis coupled with IR

- Quantification of dissolved elements: ICP/AES (Mg, Si, Fe), inorganic carbon

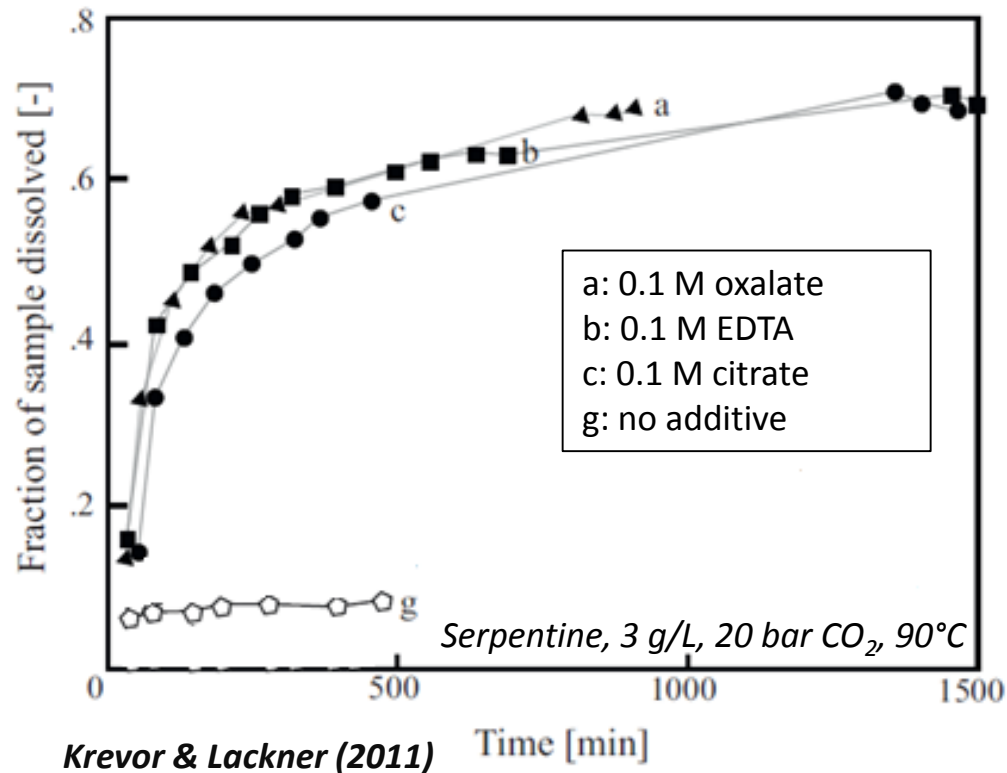
## Studied MC pathway



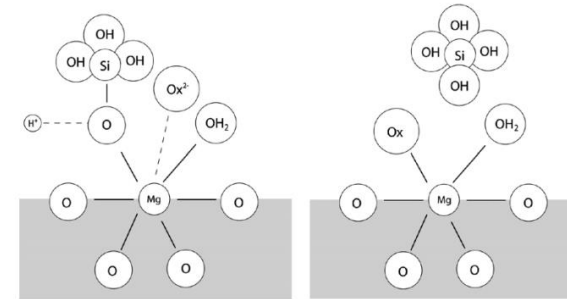
# CHELATING AGENT APPROACH

# State of the art

## ➤ Effect of organic polyacids on dissolution rate



## ➤ Mechanism



Olsen & Rimstidt (2008)

## ➤ Oxalate-enhanced dissolution rate

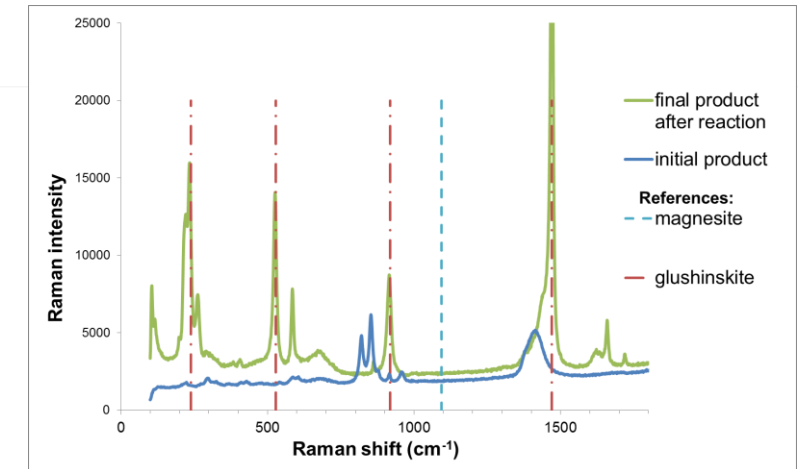
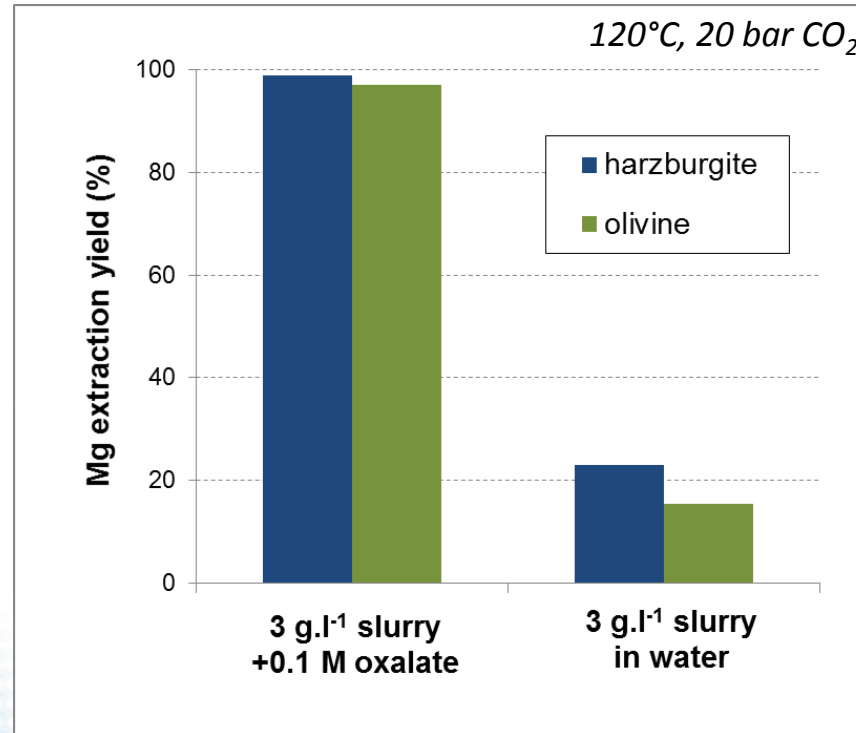
$$r = \left( \frac{1 + \beta K_x a_{ox}}{1 + K_x a_{ox}} \right) r_{H^+}$$

≈ 7 at 0.1 M oxalate (olivine, pH 5, 120°C)

Prigiobbe & Mazzotti (2011)

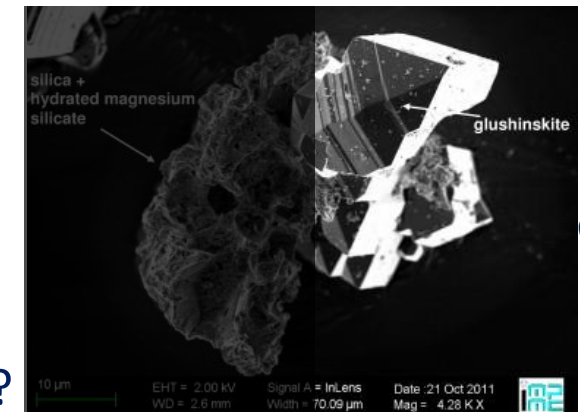


# Oxalate-enhanced dissolution of olivine: leaching yield & Mg speciation

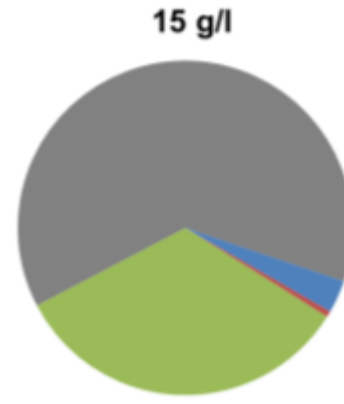
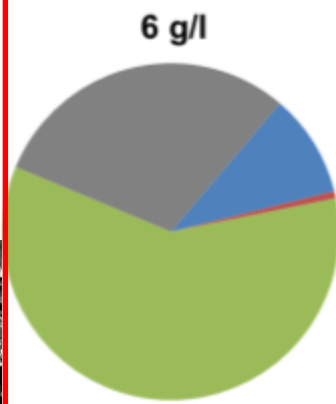
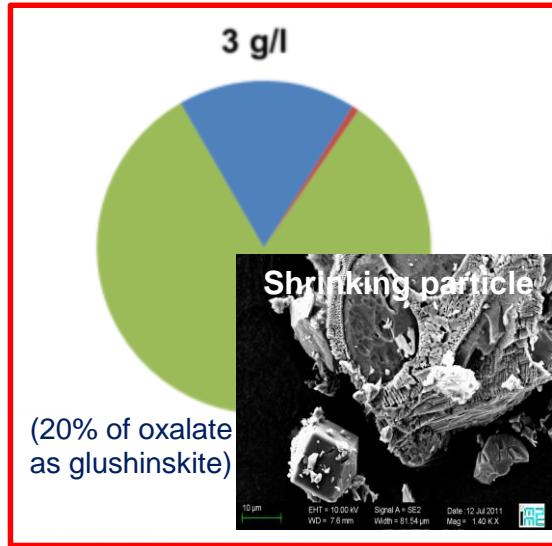


➤ Significantly improved extraction yield with oxalate at 3 g/L, but formation of solid by-product: **glushinskite** (MgC<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O)

➤ Any compromise between glushinskite precipitation & Mg carbonation?



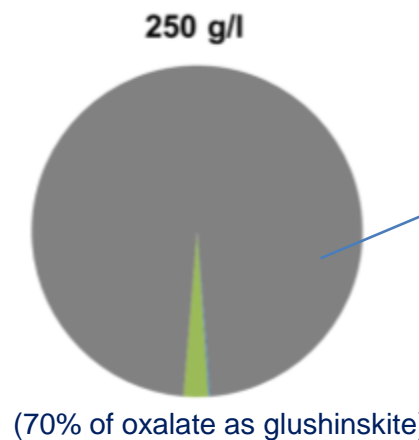
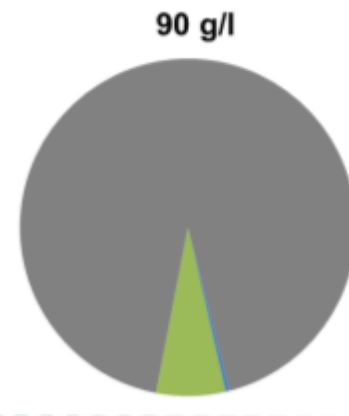
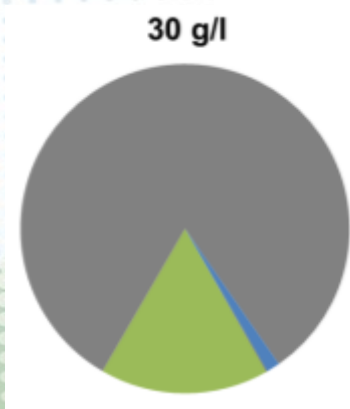
# Oxalate-enhanced dissolution of olivine: effect of slurry concentration



## Mg speciation after 24h

120°C, 20 bar CO<sub>2</sub>,  
olivine < 100 μm,  
speciation after cooling

- Carbonates
- Glushinskite
- Olivine
- Solution



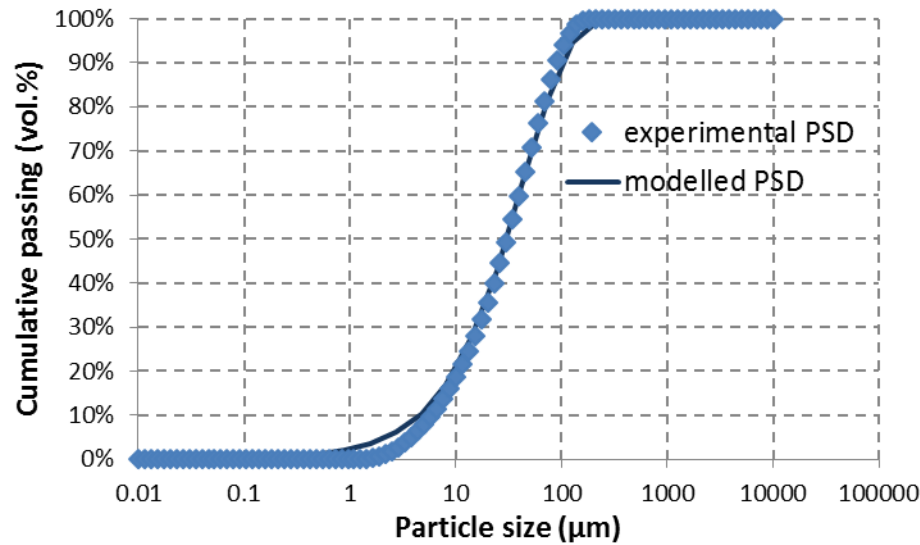
- Carbonation yield < 1% → not a viable option in these conditions
- Could geochemical modeling predict such competing effects?



# Oxalate-enhanced dissolution of olivine: kinetic modeling

Rate-limiting step: ore dissolution (instant. gas absorption & solid precipitation)

~20 size classes ( $SSA_{0i}$ ,  $m_{0i}$ ) generated from Rosin Rammler PSD model



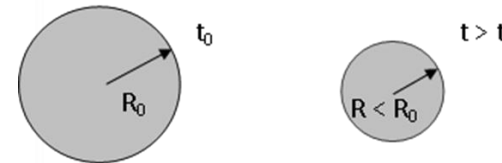
( $\alpha$  &  $d_{90}$  parameters fitted to match experimental PSD)

## Shrinking particle model

$$\frac{dn_i}{dt} = -r \cdot SSA_{0i} \cdot m_{0i} \left( \frac{n_i(t)}{n_{i0}} \right)^{2/3}$$

$$r = \left( \frac{1 + \beta K_x a_{ox}}{1 + K_x a_{ox}} \right) r_{H^+}$$

$$r_{H^+} = k_0 \cdot \exp(-E_a/RT) \cdot a_{(H^+)}^n (1 - 10^{SI})$$



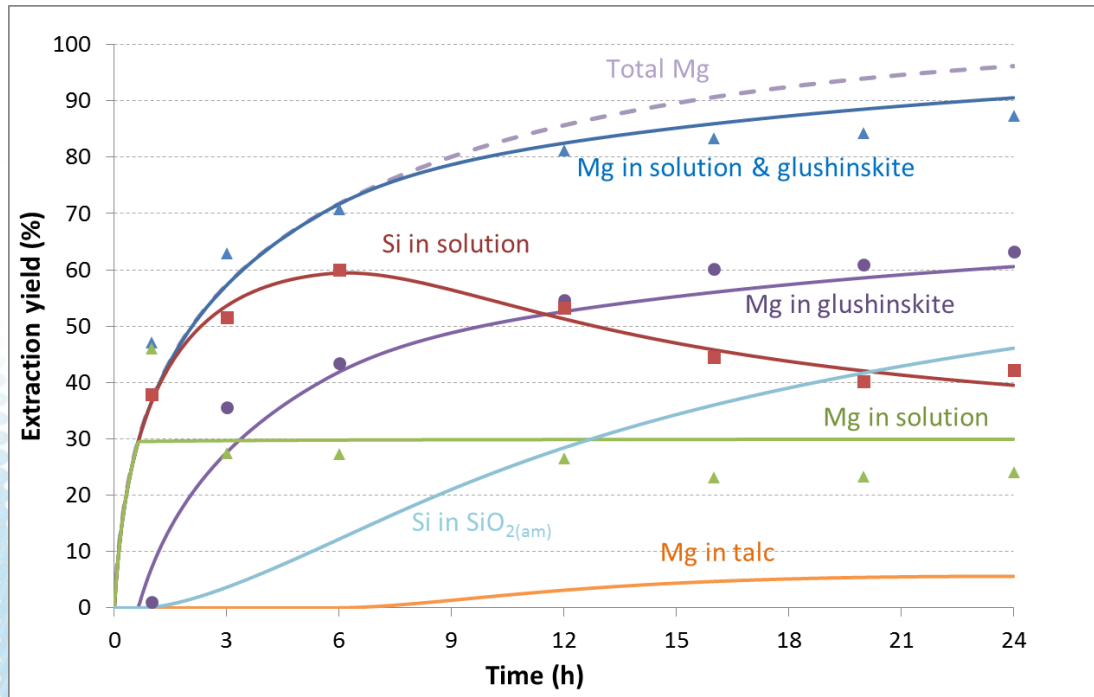
surface reaction rate from Prigiobbe & Mazzotti (2011)





## Oxalate-enhanced dissolution of olivine: geochemical modeling (with CHESS)

- Issues:**
- Mg-oxalate species missing in used thermodynamic database (CTDP)
  - Glushinskite equilibrium data only available at 25°C
- dedicated precipitation experiments at 120°C & **database updating**



*Bonfils et al. (2012)*

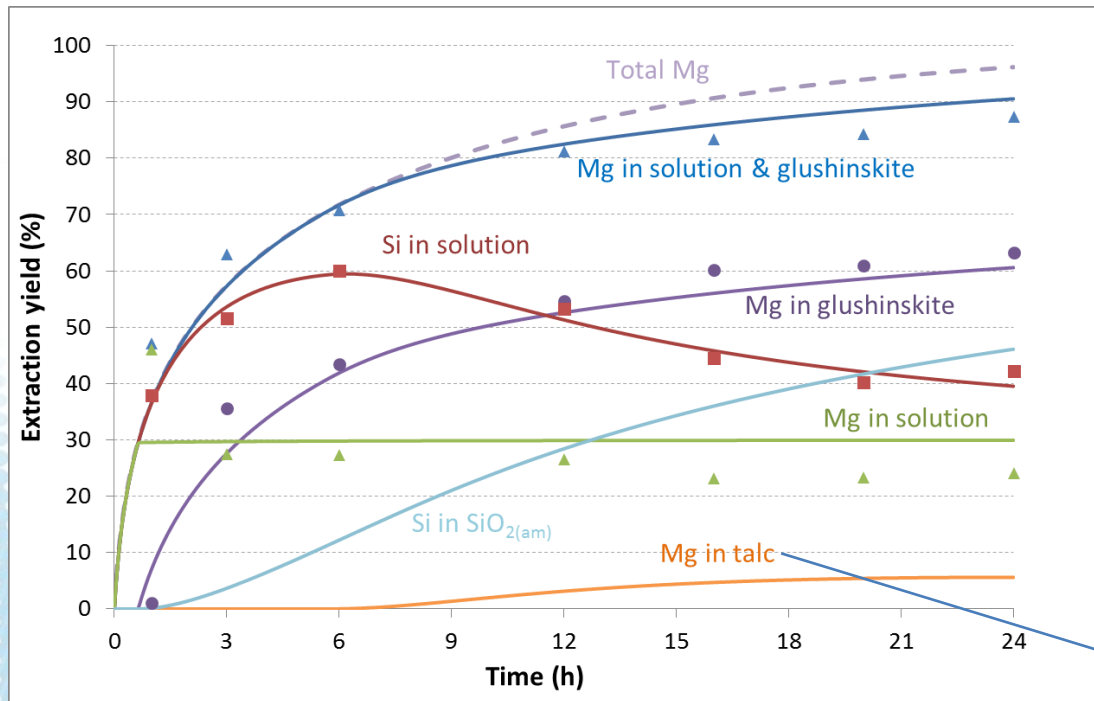
- Good agreement with experimental data at 3 g/L  
→ chemical controlled reaction

120°C, 20 bar CO<sub>2</sub>,  
3 g/L olivine < 100 μm

Simulation with CHESS  
code (*van der Lee, 2007*)  
after estimation of  
precipitation rate  
constants for silica & talc

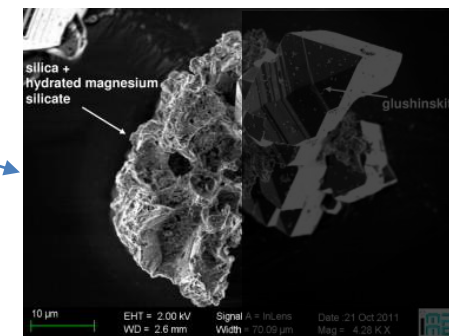
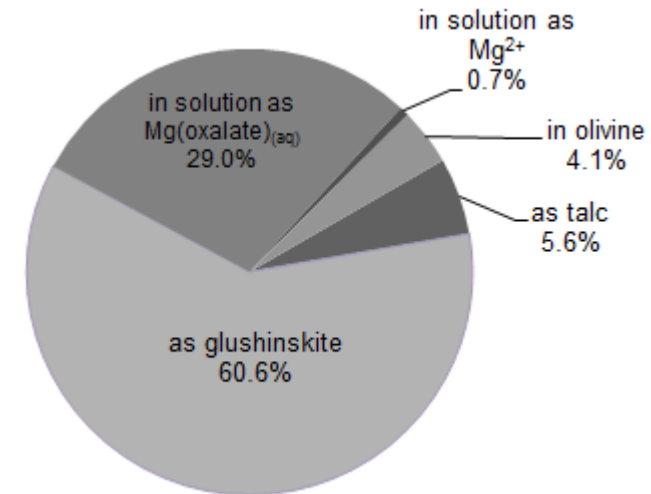
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- Issues:**
- Mg-oxalate species missing in used thermodynamic database (CTDP)
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Bonfils et al. (2012)

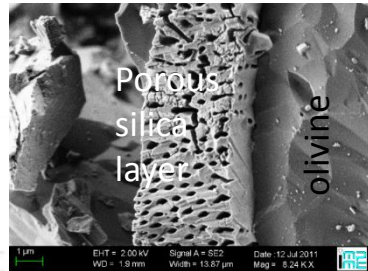
- Good agreement with experimental data at 3 g/L
- chemical controlled reaction



## Learnings & issues

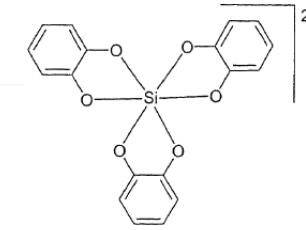
- Organic polyacid salts (oxalate, citrate, EDTA): too strong Mg binders for MC at moderate  $P_{CO_2}$
- Alternative option: silica(te) layer modulation / impeding by ligands

### Oxalate



### Catechol

Carbonation yield increased from 9.6% after 95 h (water) to 14.5% after 75 h (0.5 M catechol)



Si-catechol complex

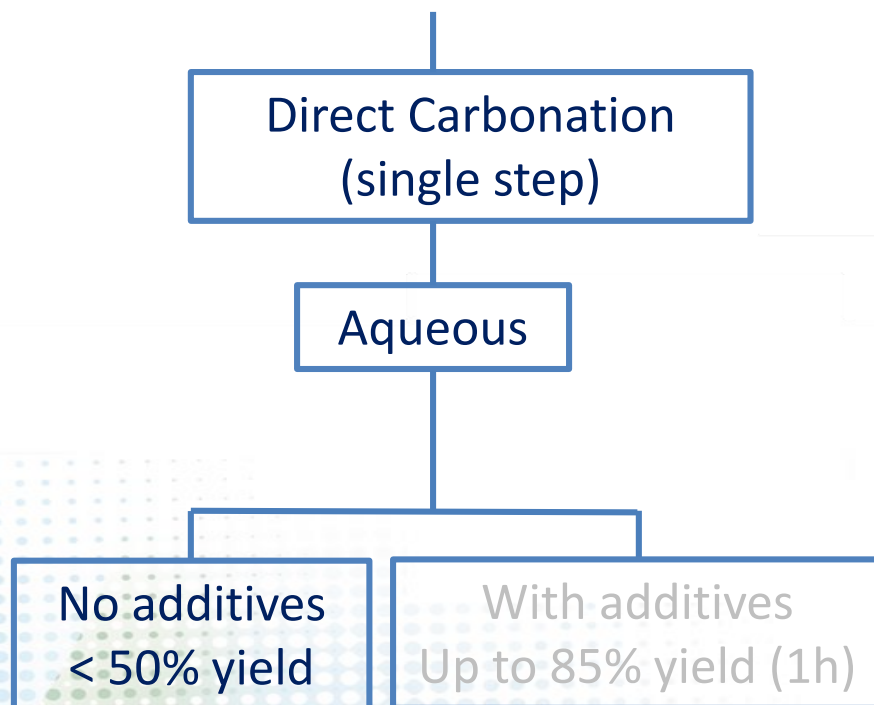
(*Barnum, 1970;*  
*Russo-Mascioli, 2001*)

but might be ore-sensitive (complexation with Fe, Al ...)

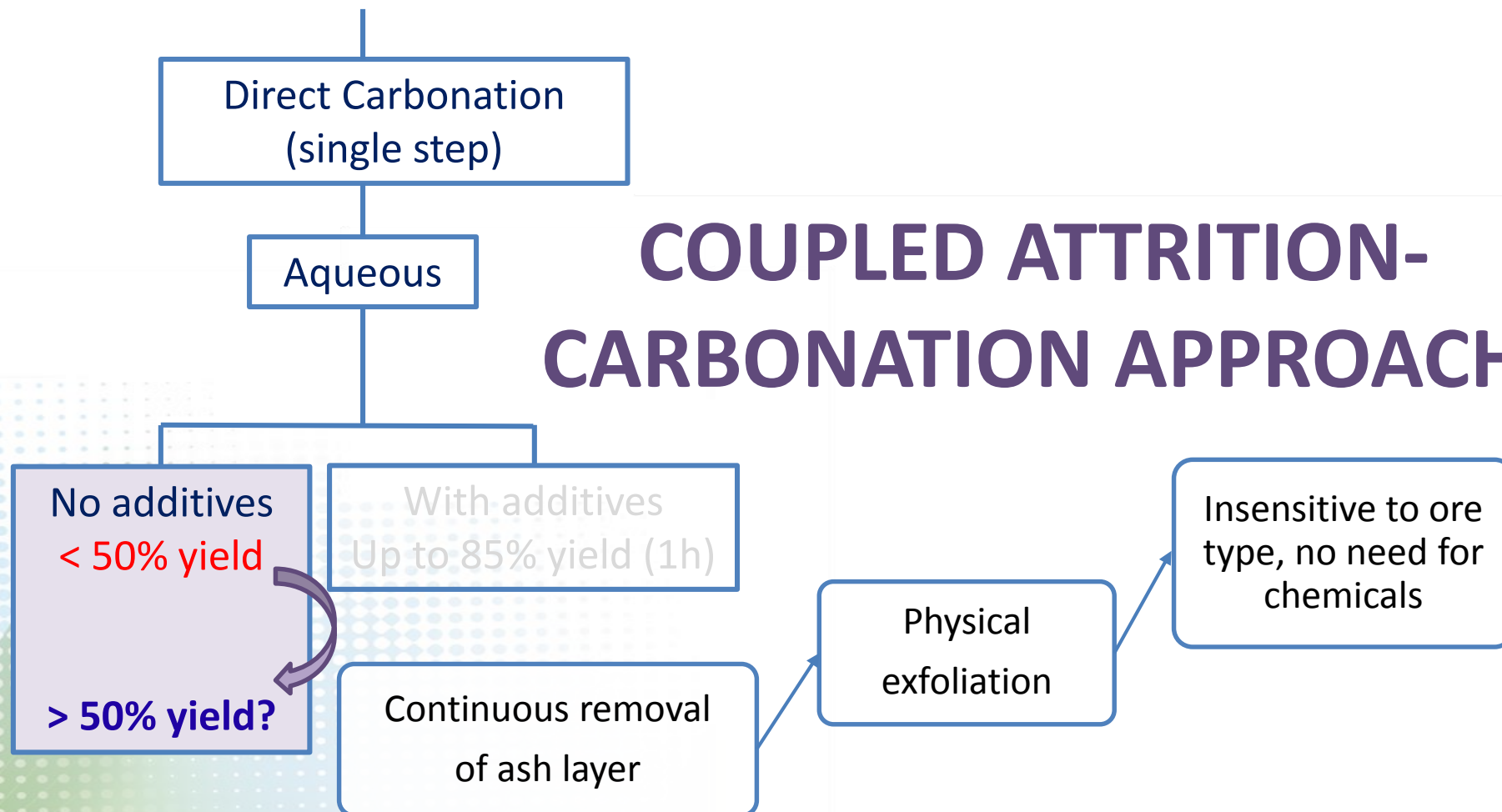
- Exhaustive analysis of both liquid & solid products = MANDATORY
- Geochemical modeling as a valuable tool to predict effect of all inputs, but
  - \* careful selection & analysis of thermodynamic database required
  - \* going from & to experimental data needed



## Studied MC pathways

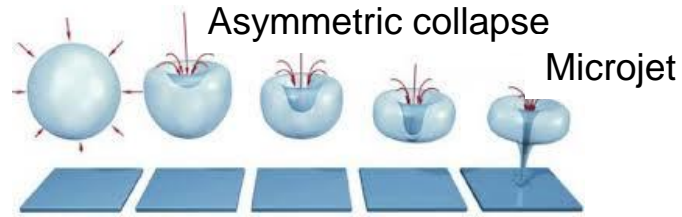


## Studied MC pathways



## State of the art

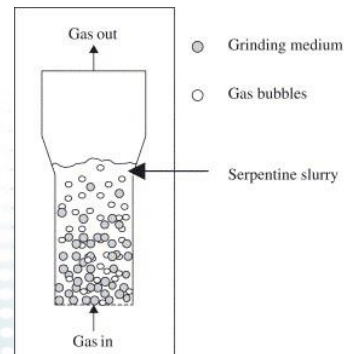
- **High power ultrasound**



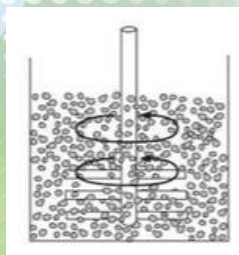
<http://eswt.net/cavitation>

**Solid surface**

- **Fluidized bed with grinding medium**



- **Stirred tank with abrasive particles**



- Enhanced carbonation yield (with quartz particles up to 60 wt.%)

*Santos et al., 2011-2013; McKelvy et al., 2004; Park & Fan, 2004*

Can be operated at moderate P (< 10 bar),  
but dampened under high T (> 50 °C)  
→ mainly in a sequential process

- Mixed results

*Park & Fan, 2004*

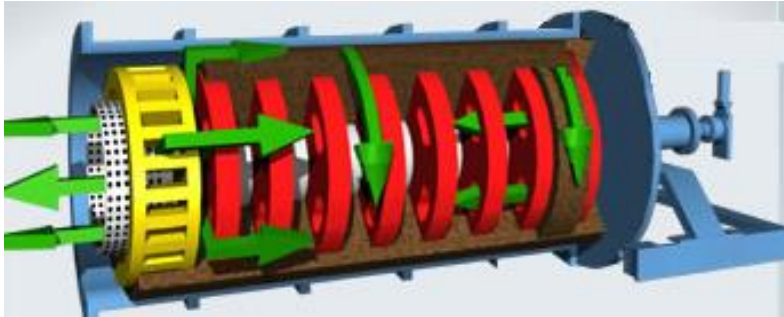
Might be difficult to control (density and/or size differences between reactive & inert particles),  
limited amount of grinding medium (20 wt.%)

- Some improvement of ore dissolution

*Chizmeshya et al., 2007*



## Proposed technical solution: stirred ball mill



IsaMill™ (Netzsch)

### Advantages:

- ✓ proven technology at large scale
- ✓ slurry conc.: up to 40%
- ✓ feed PSD: from  $\mu\text{m}$  to mm size range
- ✓ operability under high T & high P
- ✓ scalability from 4 L to 50 m<sup>3</sup>

### Issues:

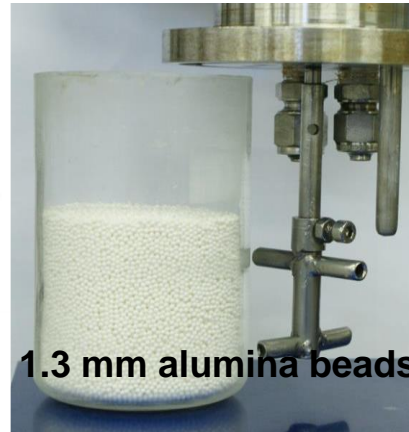
- (long-term) exfoliation efficiency?  
*significant carbonation yield?*  
*within a reasonable solid residence time?*
- passivation layer attrition vs. breakage of ore particles?

↓

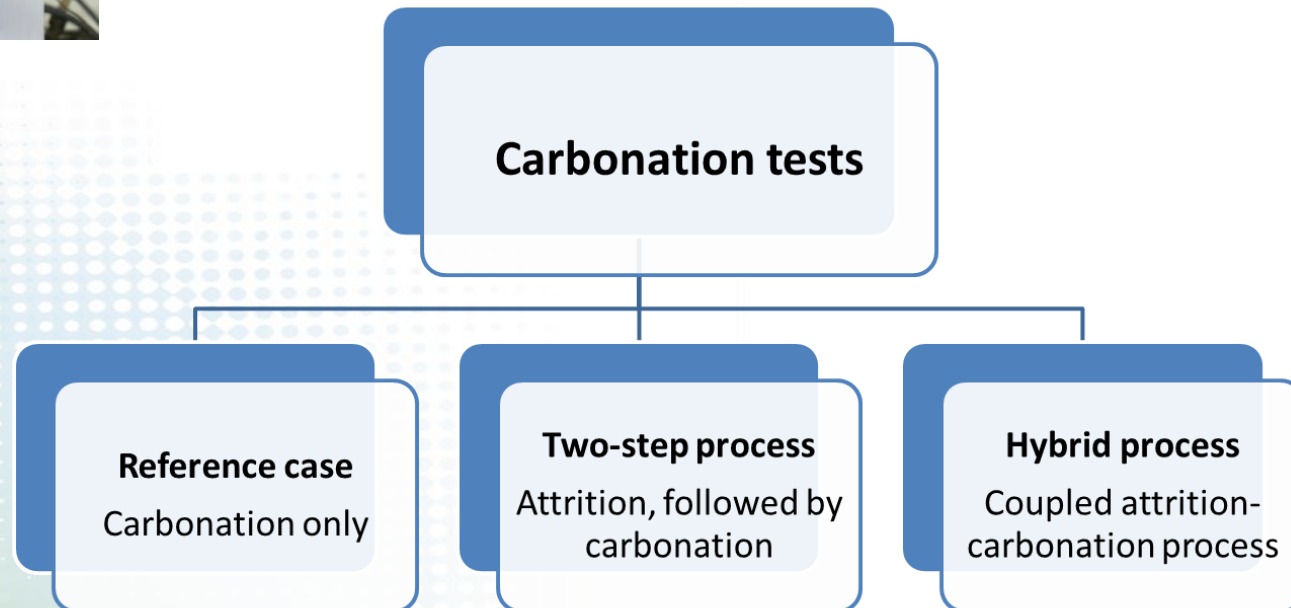
energy efficiency: matching attrition process with passivation process?

# Proof of concept

**Grinding media:** 90 mL of 1-2 mm beads  
 + 80 mL of slurry (90-250 g/L) –  $\omega = 800$  rpm



1.25-1.6 mm sand particles (99% SiO<sub>2</sub>)

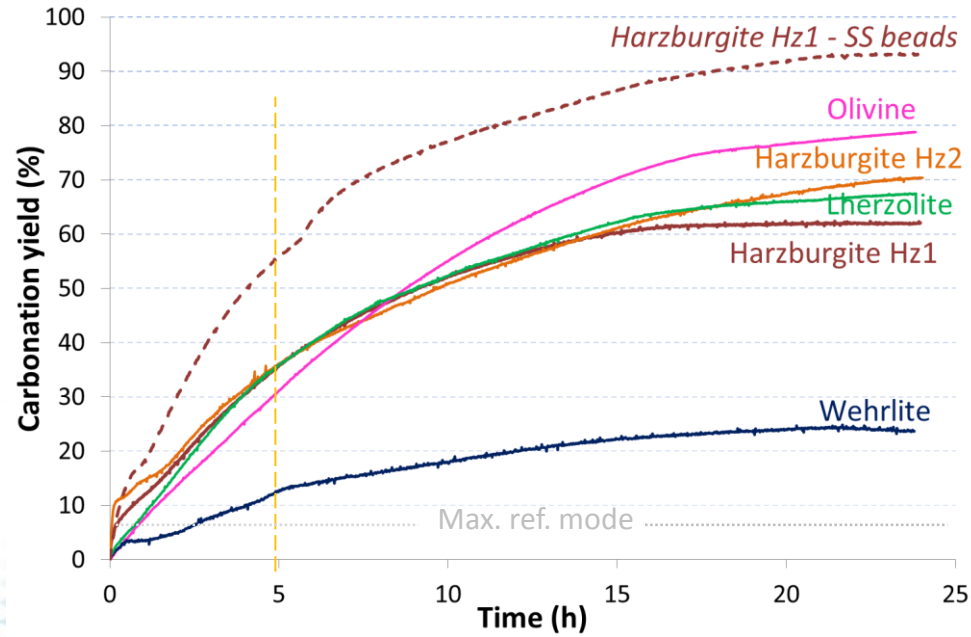






# Proof of concept

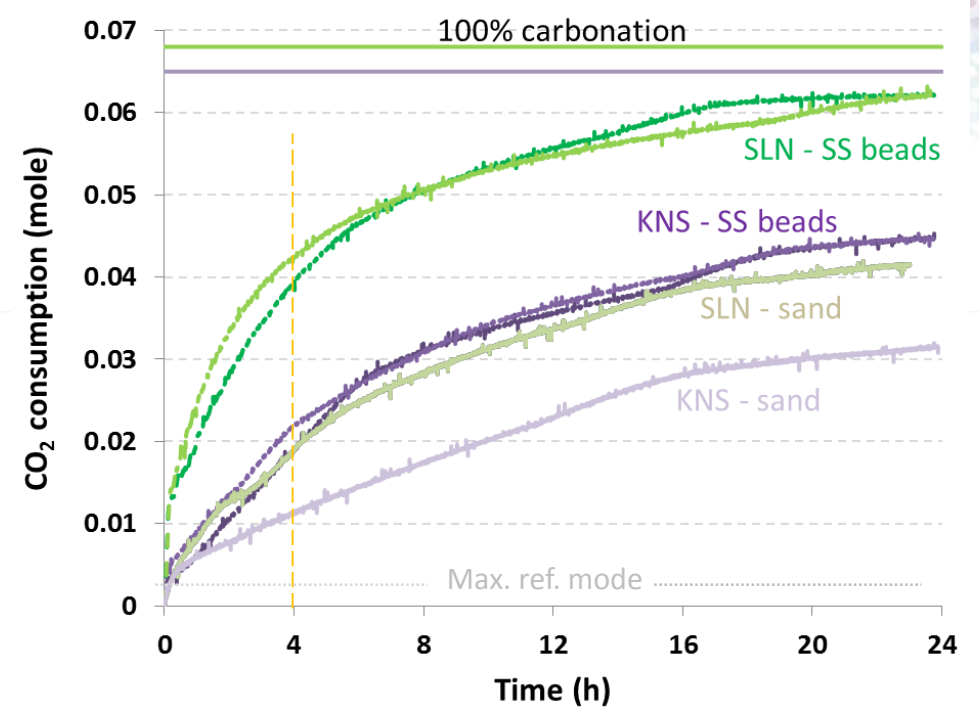
## Ores



Extent of carbonation for different ore types  
(180°C, 20 bar CO<sub>2</sub>, ore conc.: 90 g/L,  
1-2 mm Al<sub>2</sub>O<sub>3</sub> — or SS grinding media ---)

- Significant extent of carbonation  
(vs. < 8% in 24 h without attrition)
- Almost insensitive to ore type
- Noticeable influence of grinding medium

## Slags



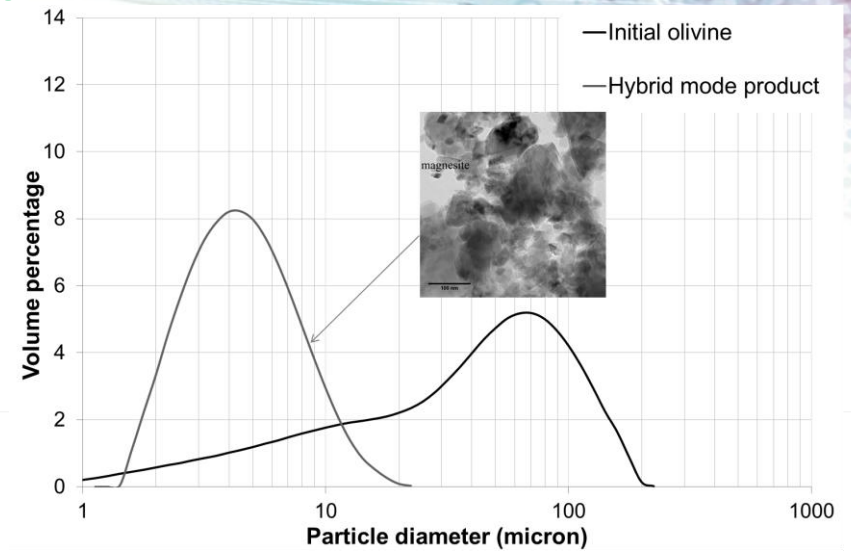
Extent of carbonation for different slag types  
(180°C, 20 bar CO<sub>2</sub>, ore conc.: 90 g/L,  
1-2 mm sand — or SS grinding media ---)





## Proof of concept

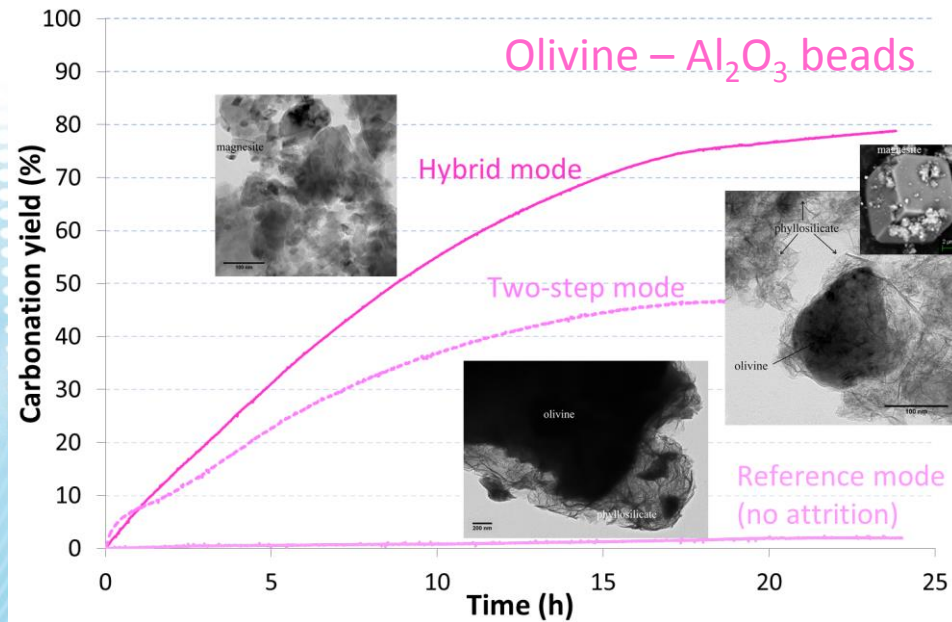
Size effect only?



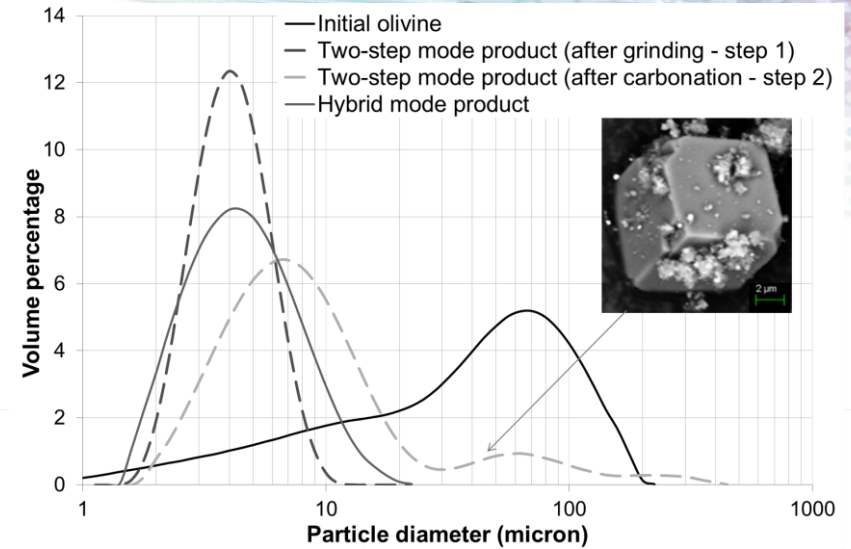
## Proof of concept

### Size effect only?

➤ Proof of synergy between attrition and leaching



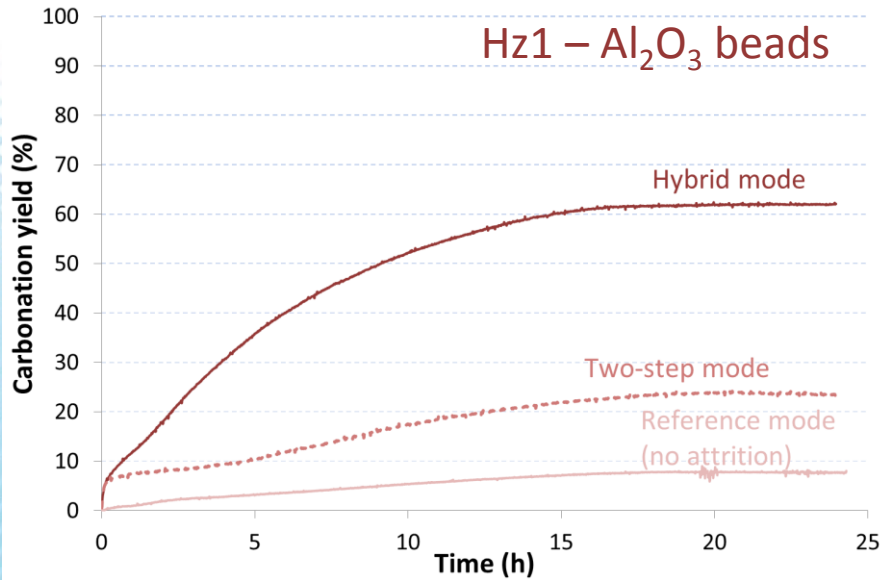
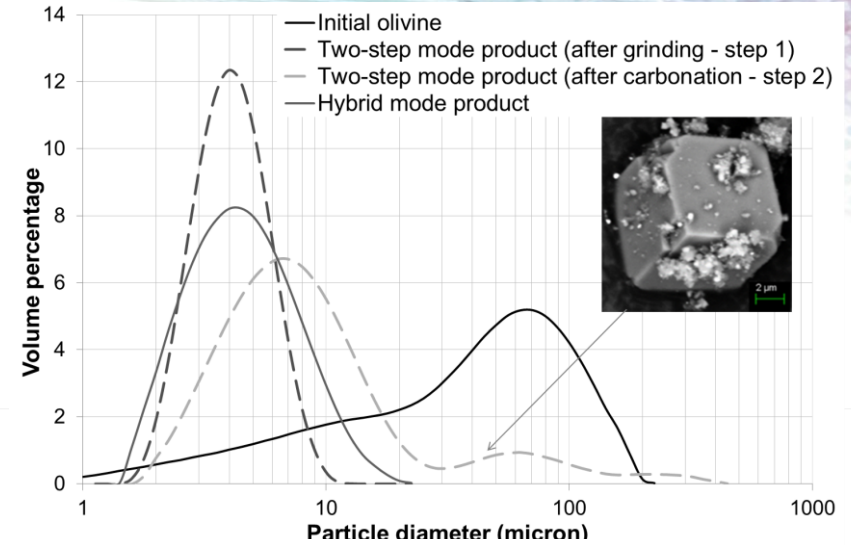
180°C, 20 bar CO<sub>2</sub>, ore conc.: 90 g/L



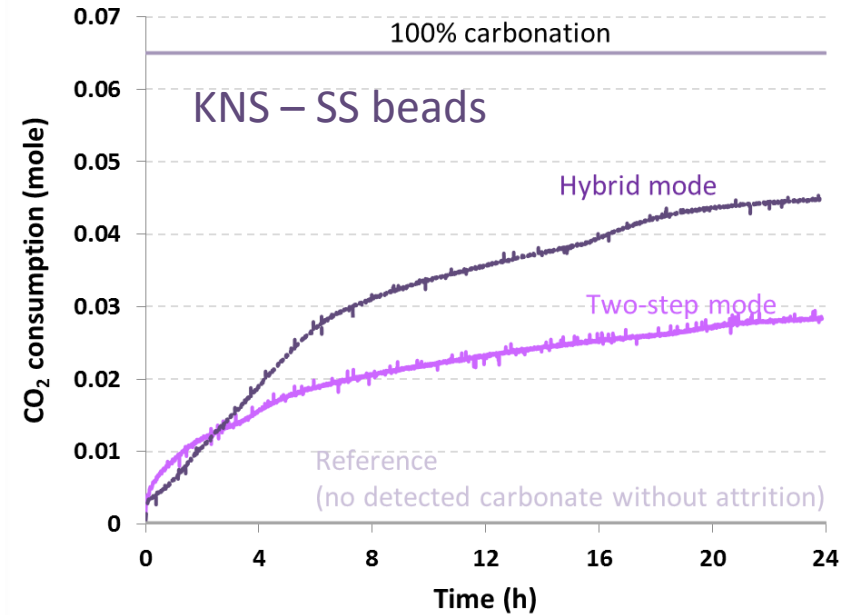
## Proof of concept

### Size effect only?

➤ Proof of synergy between attrition and leaching



180°C, 20 bar CO<sub>2</sub>, ore conc.: 90 g/L





## Life Cycle Assessment (LCA)

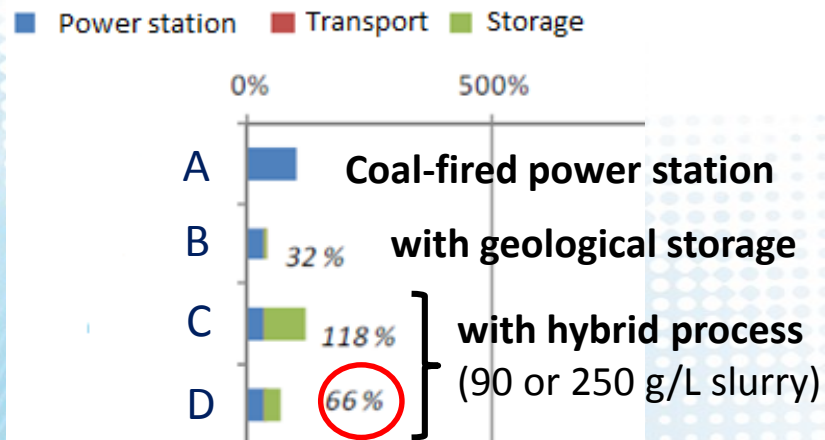
**Functional unit:** 1 MWhe with a coal-fired power station

**Main hypotheses:**

- yield of ~ 80% in 24 h at 20 bar CO<sub>2</sub> & 180°C
- CO<sub>2</sub> pipeline transport over a 300 km distance
- no recycling of process solution; no valorization of products

**Impacts accounted for:**

- CO<sub>2</sub> capture & compression
- crushing & milling of ore from 1 cm down to 100 μm
- mechanical energy expended for attrition
- reactants pre-heating & cooling (after heat integration)

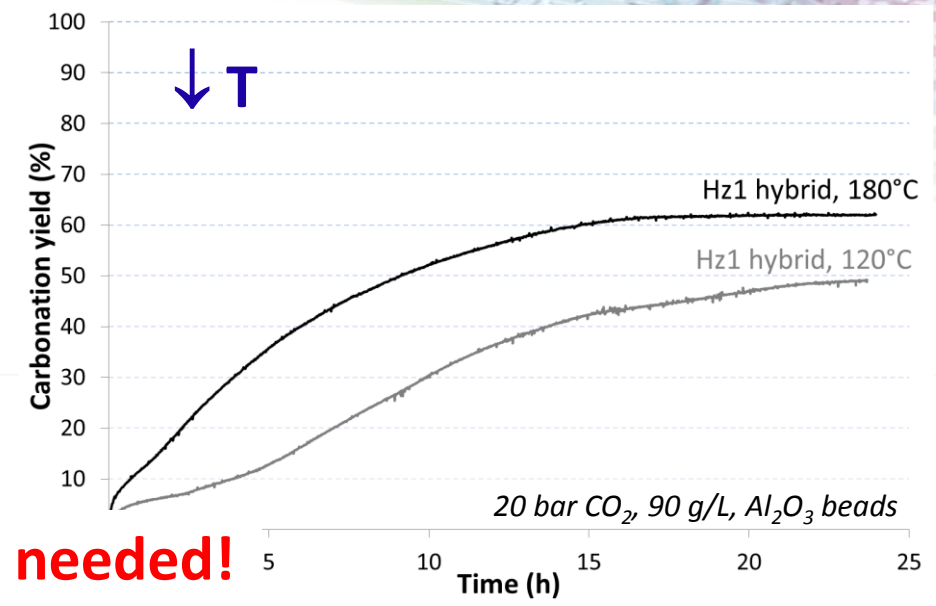
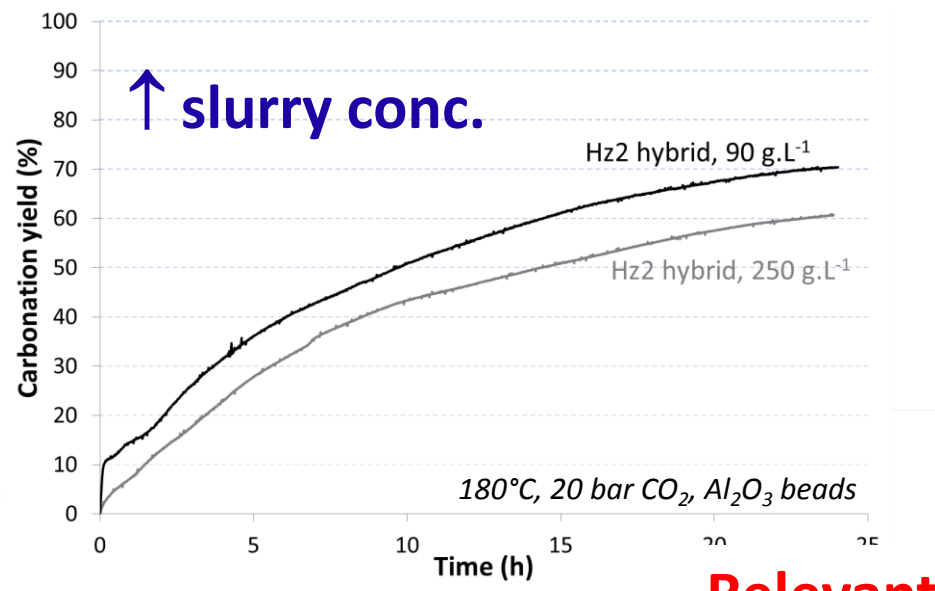


Net global warming potential (kg CO<sub>2</sub>-eq)

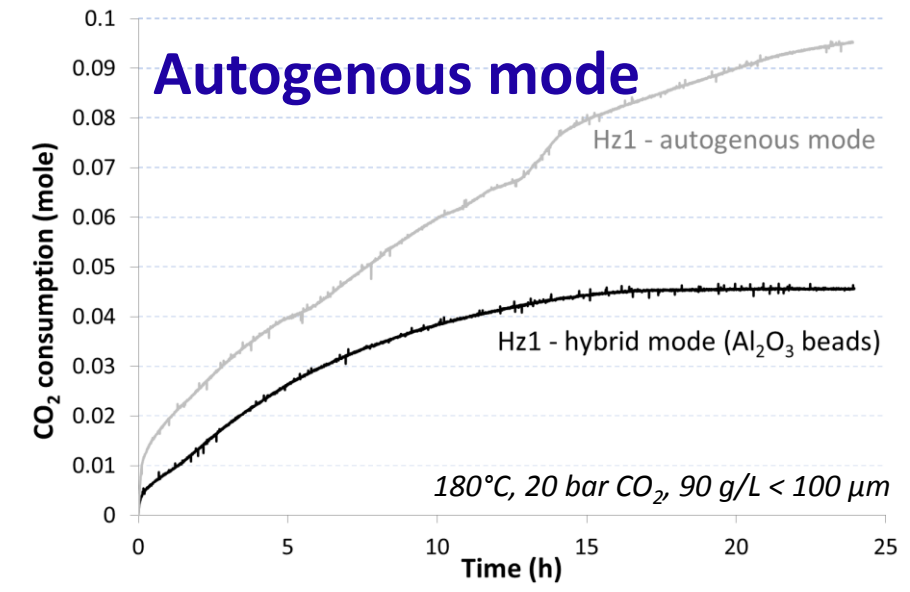
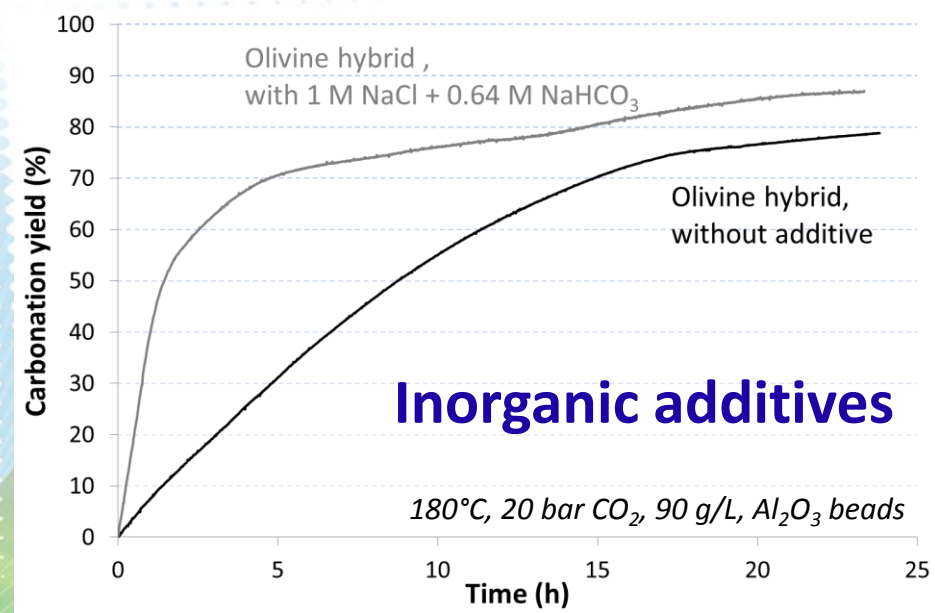
- Promising results regarding CO<sub>2</sub> avoided, without any process optimization
- Beneficiation of products & water recycling will also improve other LCA criteria (natural resource depletion +110% for case D)



# Possible ways of optimization



**Relevant model is needed!**

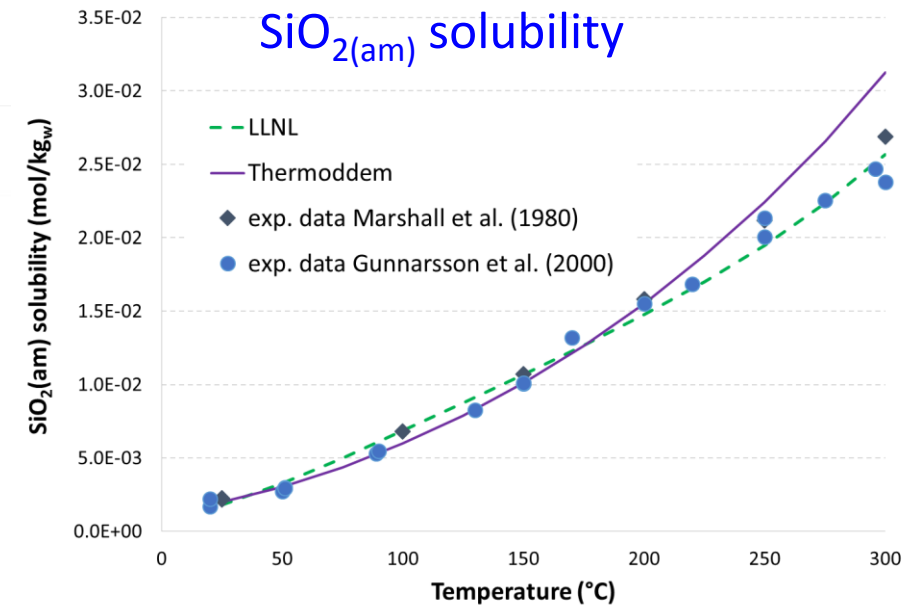
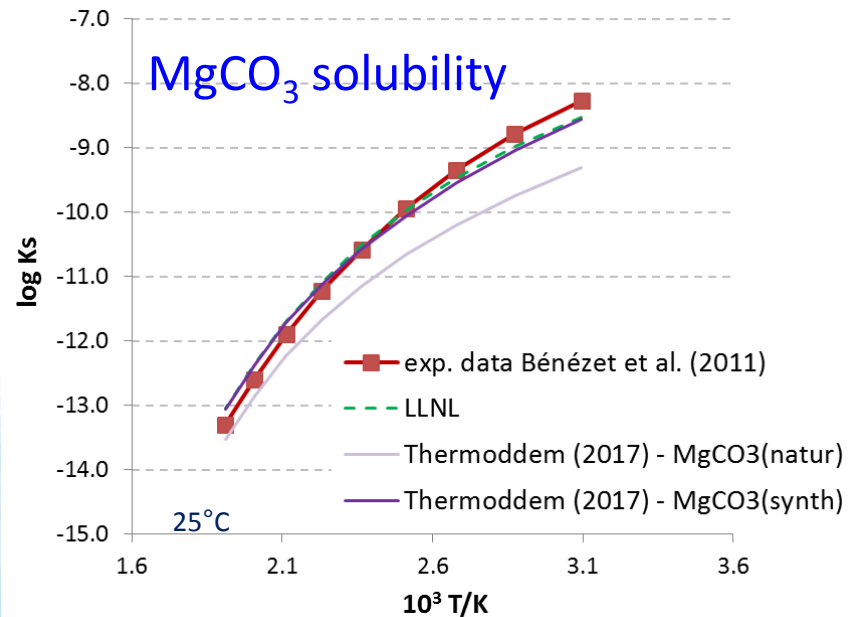


# Geochemical modeling as a process design tool

Geochemical code: PHREEQC v.3 (Parkhurst & Appelo, 1999, 2013)

Available databases: LLNL, Thermoddem

*Selection based on existing exp. data  
for main system components*



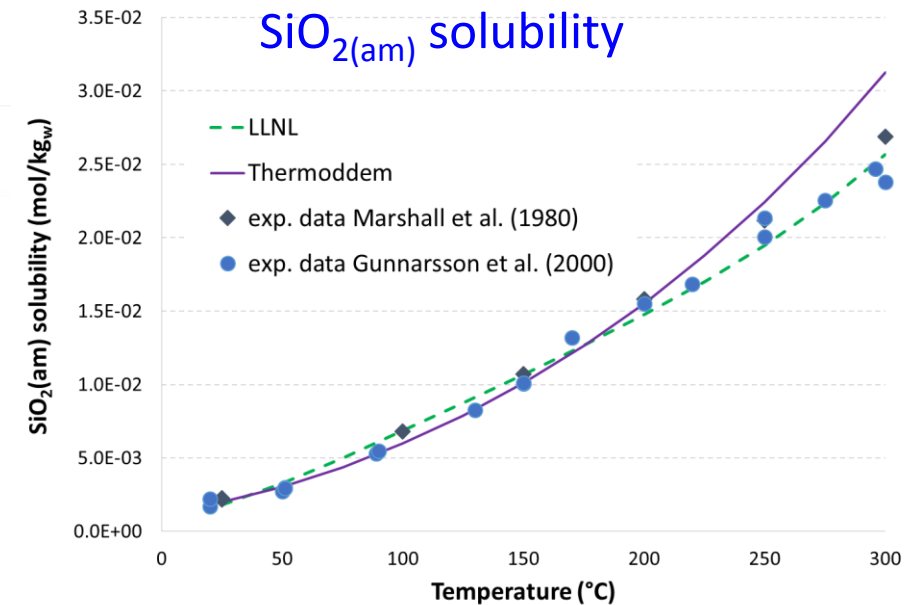
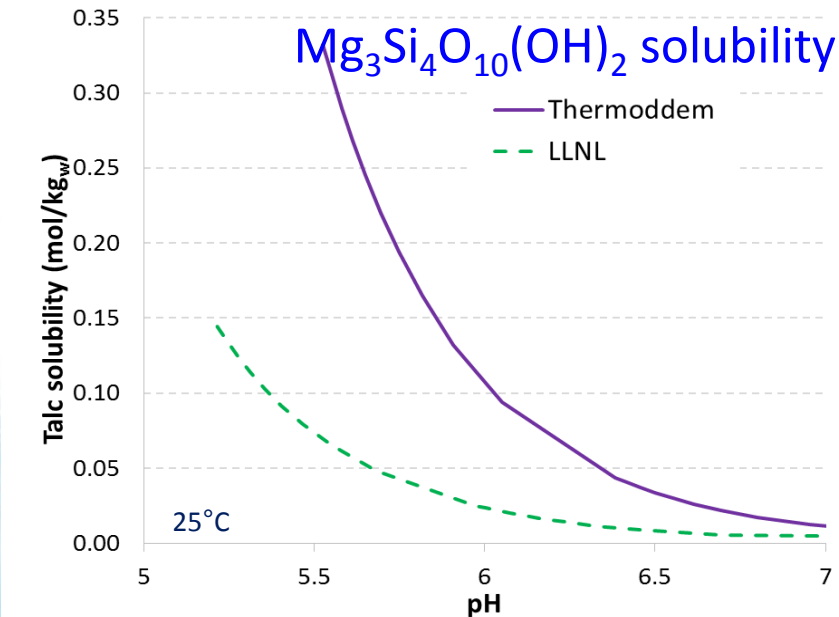


## Geochemical modeling as a process design tool

Geochemical code: **PHREEQC v.3** (Parkhurst & Appelo, 1999, 2013)

Available databases: LLNL, Thermoddem

*Selection based on existing exp. data  
for main system components*



Discrepancies for talc, but no solubility data available (in similar conditions) → assessment on carbonation results

**Liquid:** extended Debye-Hückel activity coefficient model (low salinity)

**Gas:** Peng-Robinson equation of state (non-ideal behavior of CO<sub>2</sub> - H<sub>2</sub>O mixture at investigated P)

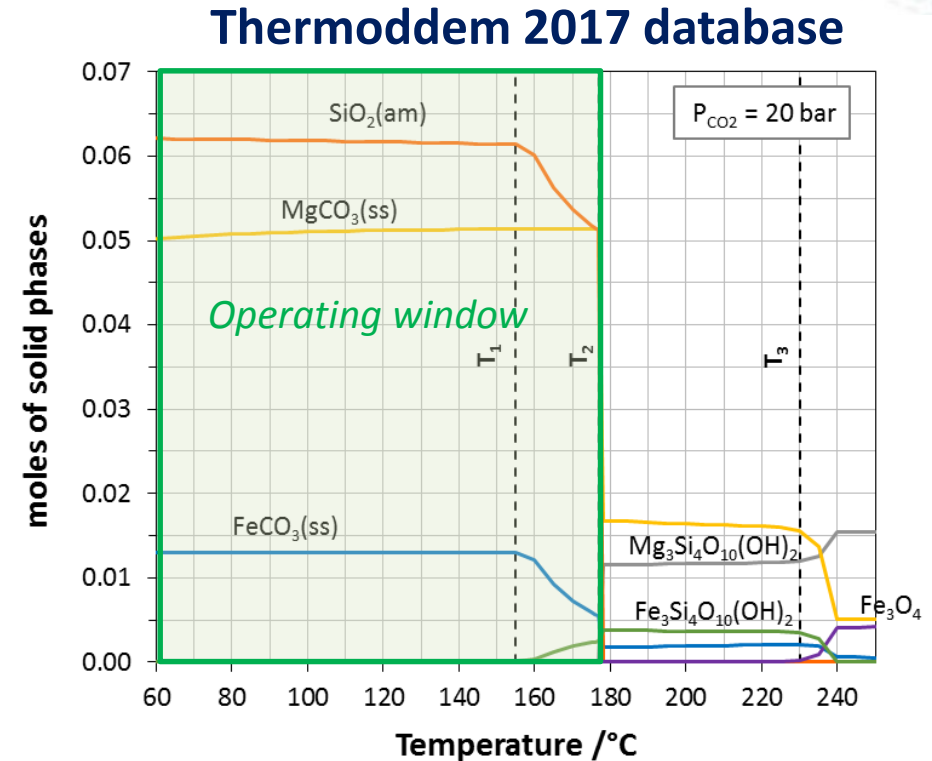
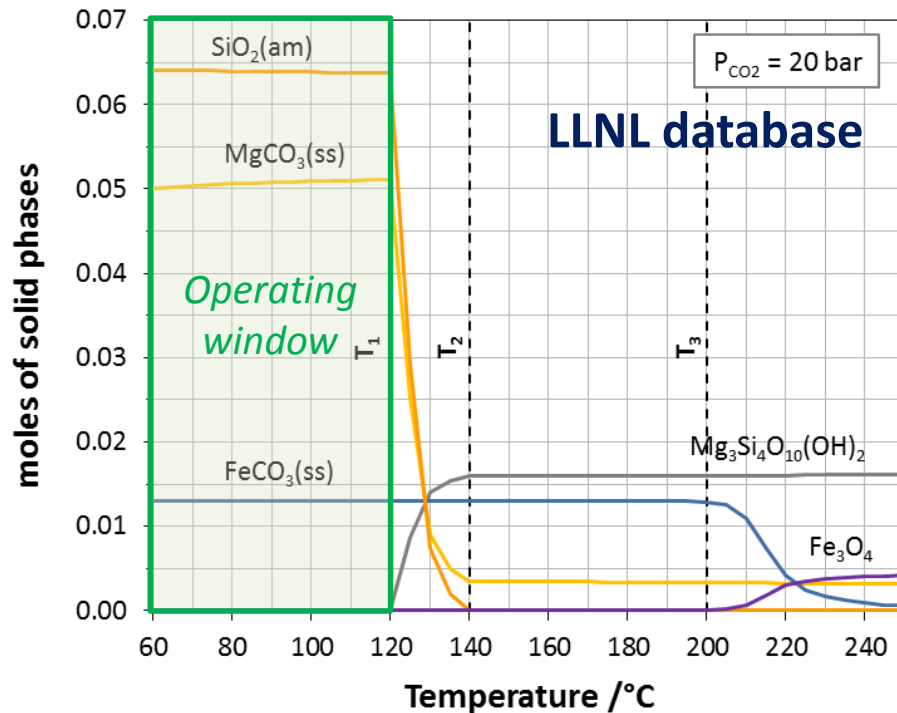
**Solid:** description of several **solid solutions** (e.g. (Mg,Fe)CO<sub>3</sub>)

## Case study 1: KNS / “inert” grinding medium

“KNS” (90 g/L) described as an assemblage of MgO, SiO<sub>2</sub> and FeO

All minerals including Mg, Si or Fe are allowed to precipitate, except quartz

### Mineral speciation of KNS-H<sub>2</sub>O-CO<sub>2</sub>



➤ Threshold T for quantitative carbonation at 20 bar CO<sub>2</sub> depends on the database!

➤ At T = 180°C and P<sub>CO<sub>2</sub></sub> = 20 bar:

~~LLNL → theoretical max carbonation yield: 25%~~

**Thermoddem 2017 → conditions close to the drop in carbonation yield**

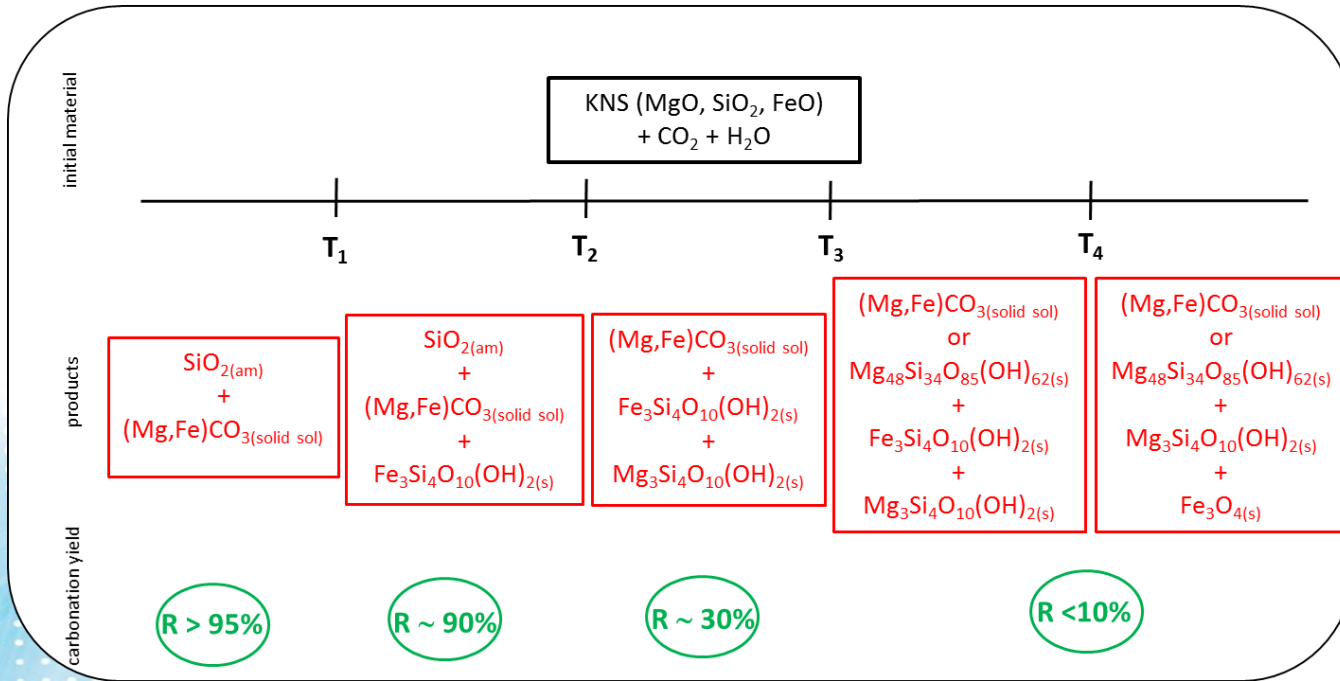


## Case study 1: KNS / “inert” grinding medium

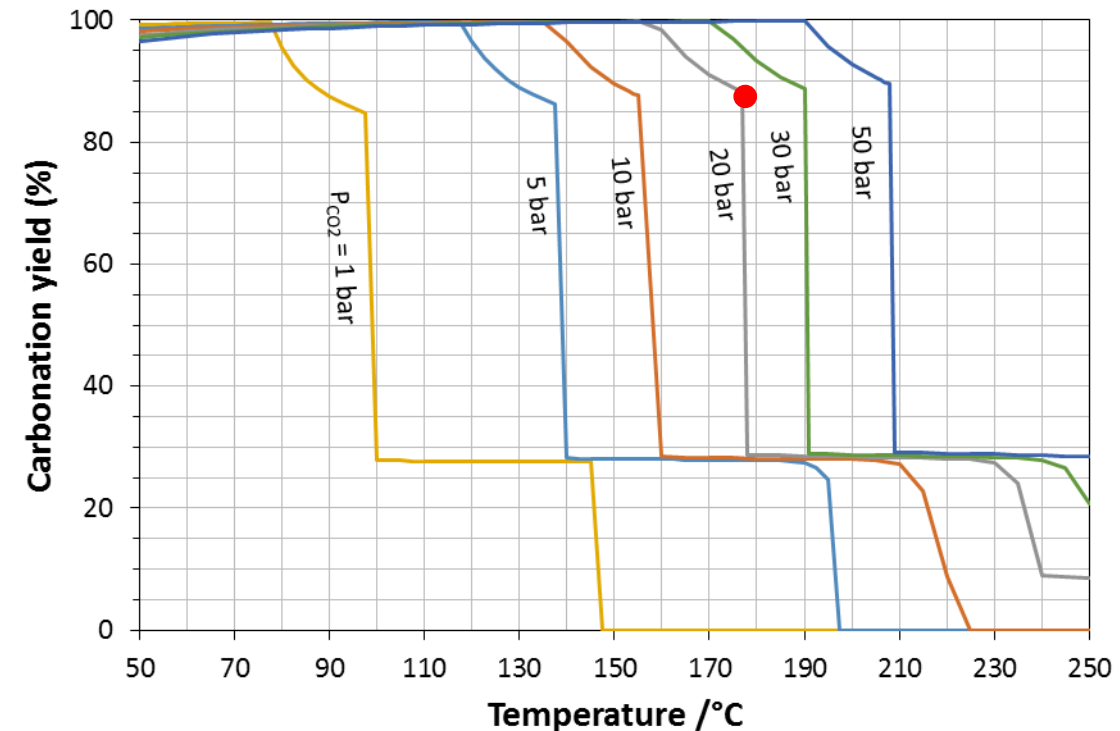
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### Mineral speciation of KNS-H<sub>2</sub>O-CO<sub>2</sub>



### Thermodem 2017



➤ Operating threshold temperature depends on P<sub>CO<sub>2</sub></sub>

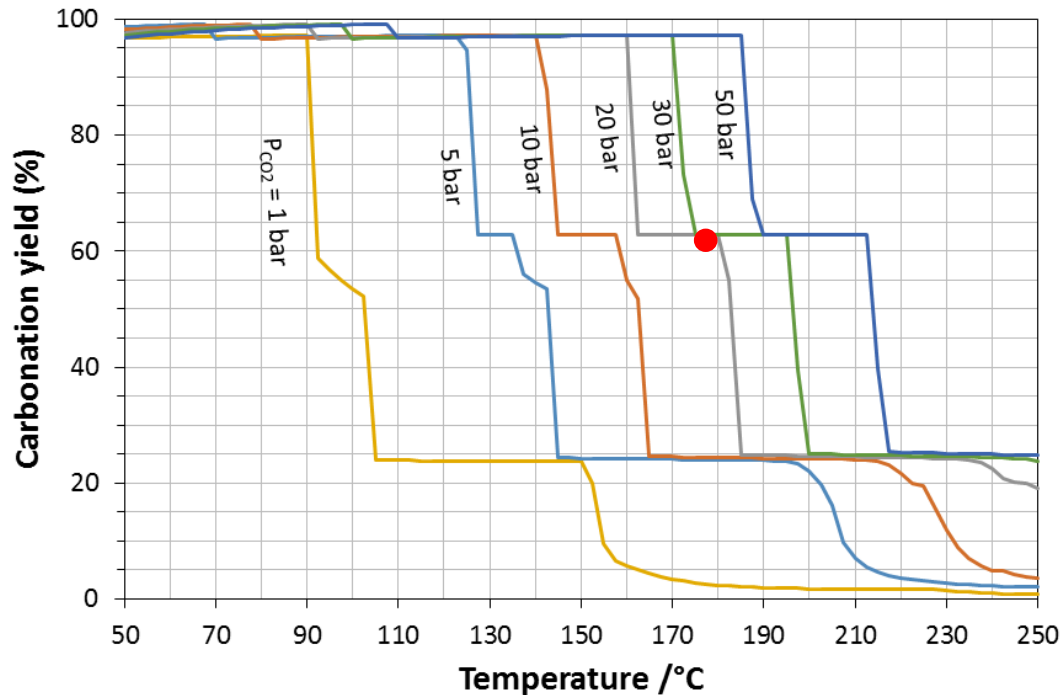




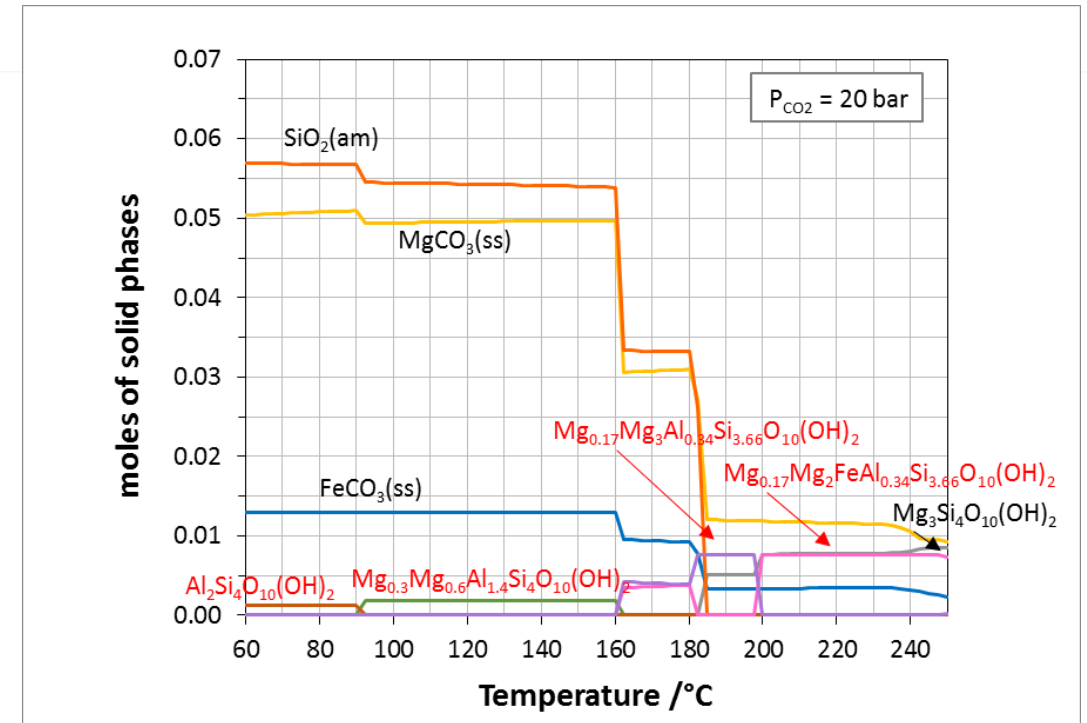
## Case study 1: KNS / “inert” grinding medium

“KNS” (90 g/L) described as an assemblage of MgO, SiO<sub>2</sub>, FeO, CaO, Al<sub>2</sub>O<sub>3</sub> and MnO  
 All minerals including Mg, Si, Fe, **Ca, Al or Mn** allowed to precipitate (except quartz)

### Mineral speciation of KNS-H<sub>2</sub>O-CO<sub>2</sub>



### Thermodem 2017



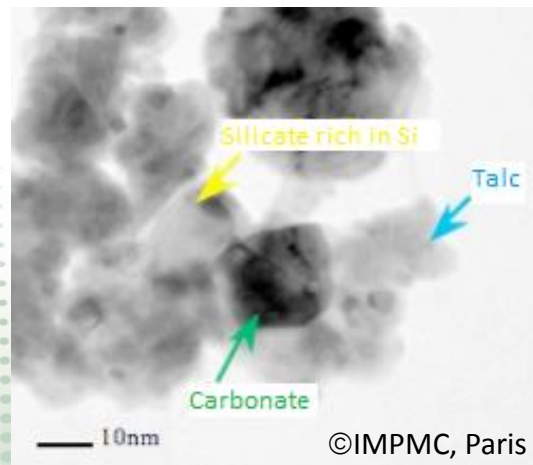
- The model predicts a noticeable effect of slag “impurities” (Al<sub>2</sub>O<sub>3</sub> ~ 2%) due to the existence of various stable aluminosilicate phases



## Case study 2: KNS / effect of grinding medium

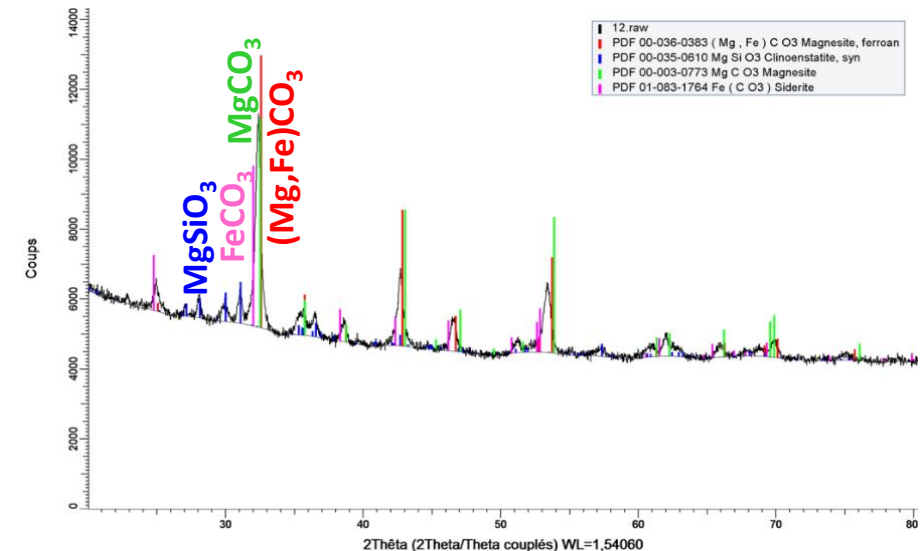
### Experimental data

- Carbonation yield ~ 50% with sand (180°C, 20 bar, 90 g/L KNS)
- Crystallized phases = initial mineral phases (enstatite, ferrosilite, augite), quartz (sand) & mixed carbonates (dominated by  $\text{MgCO}_3$  pole)
- TEM/EDX



→ Precipitation of various (amorphous) silicates

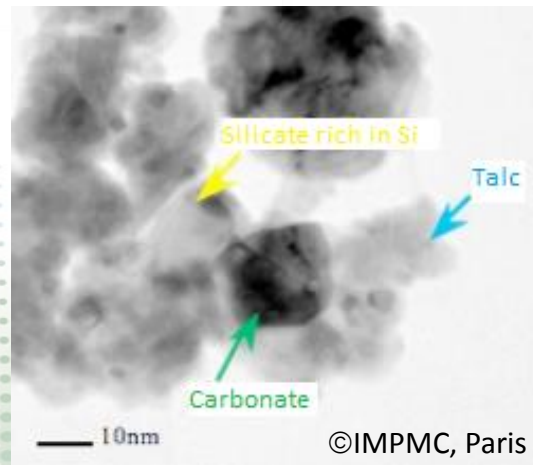
- Higher yield achieved with stainless steel beads  
... but increase of Fe content in solid product &



## Case study 2: KNS / effect of grinding medium

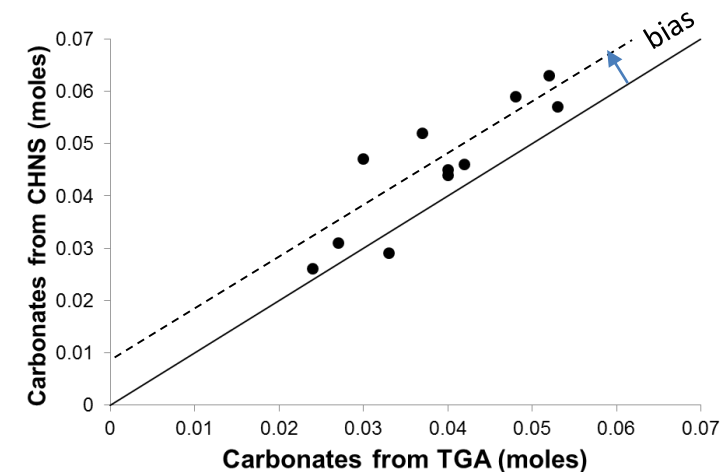
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- TEM/EDX



→ Precipitation of various (amorphous) silicates

- Higher yield achieved with stainless steel beads ... but increase of Fe content in solid product & a few discrepancies between carbonate amounts calculated from TGA and carbon content

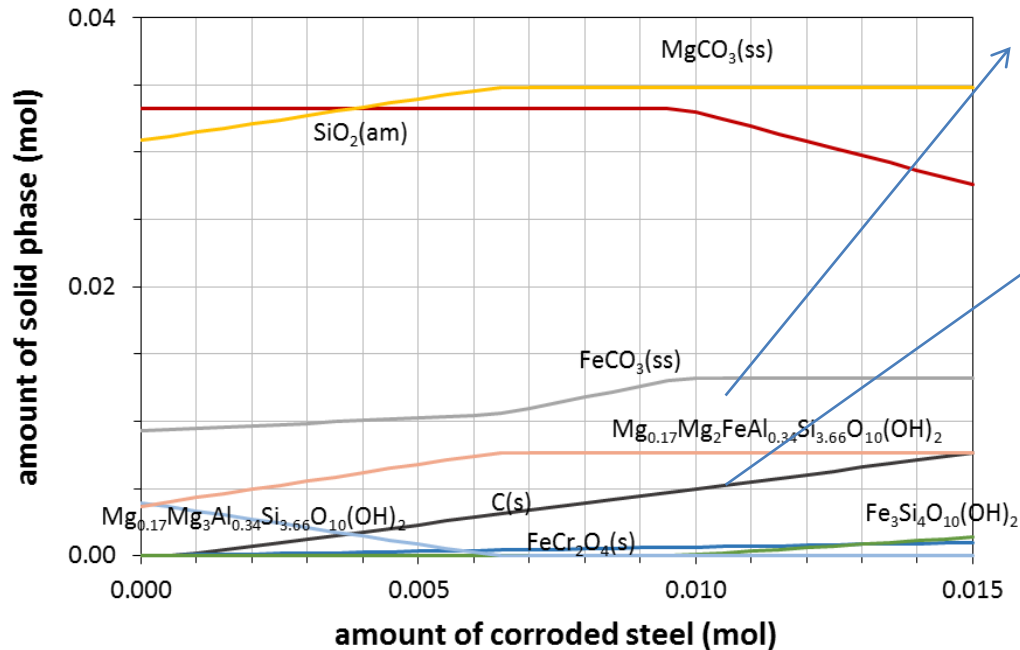




## Case study 2: KNS / effect of grinding medium

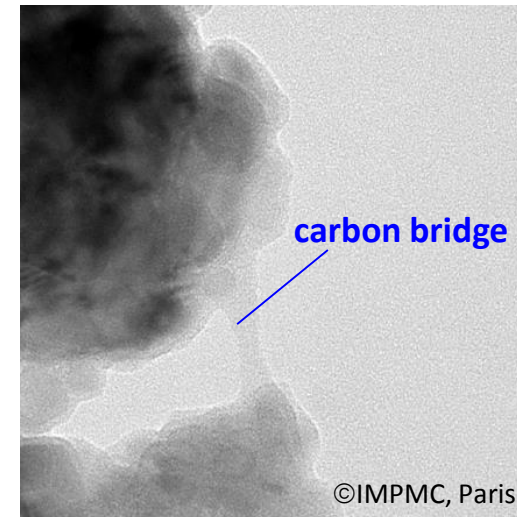
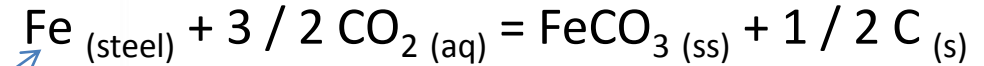
“KNS” described as an assemblage of MgO, SiO<sub>2</sub>, FeO, CaO, Al<sub>2</sub>O<sub>3</sub> and MnO  
**Steel beads** modeled as a (Fe<sub>0.87</sub>Cr<sub>0.13</sub>) solid solution

### Mineral speciation of KNS-H<sub>2</sub>O-CO<sub>2</sub>



Fe extracted from beads as FeCO<sub>3</sub> & ferro-magnesium-aluminosilicate

Formation of solid carbon:



$P_{\text{CO}_2} = 20 \text{ bar}, T = 175^\circ\text{C}, \text{slag conc.} = 90 \text{ g/L}$

➤ **Stainless steel beads are corroded during attrition-leaching under CO<sub>2</sub>**

## Case study 3: Batch simulation for olivine ore

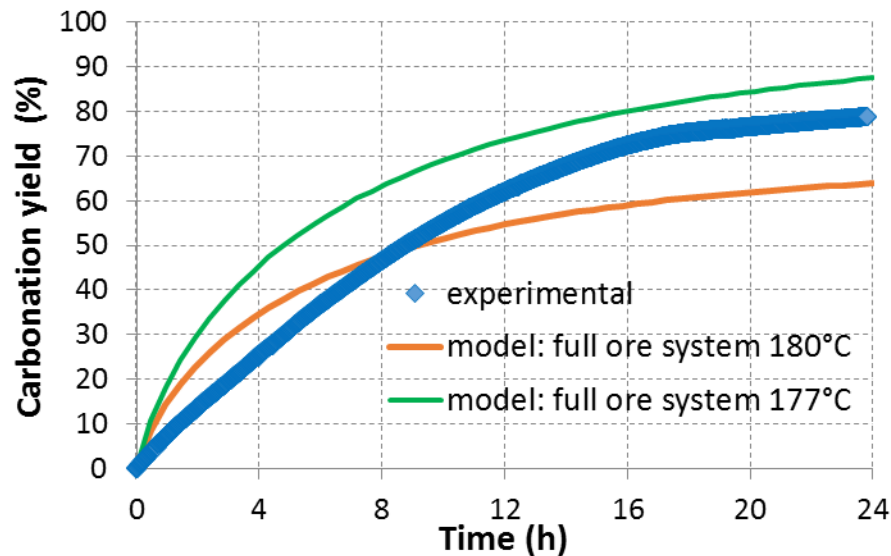
“Synthetic olivine” described as an assemblage of MgO, SiO<sub>2</sub>, FeO, CaO, Al<sub>2</sub>O<sub>3</sub> and MnO

**Initial PSD accounted for**, dissolution kinetic parameters from Prigiobbe et al. (2009)

**Alumina beads** modeled as corundum, kinetic parameters from Palandri & Kharata (2004)

### Time-evolution of carbonation yield

“Inert” grinding medium



$P_{CO_2} = 20 \text{ bar}$ ,  $T = 180^\circ\text{C}$ , ore conc. = 90 g/L

Effect of alumina grinding beads

Very slow dissolution kinetics

→ Negligible effect on system speciation

- Good agreement between experimental data and modeling
- **Process dynamics driven by the dissolution rate of fresh ore surface**



## Learnings & issues

- Proof of concept of the attrition-carbonation process with a stirred bead mill; **Synergy between attrition & carbonation**
- **Favorable LCA**; several optimization levers to improve process efficiency, cost & environmental impact
- **Grinding medium to be carefully selected (autogenous mode?)**
- **Geochemical modeling is a powerful tool** for designing the attrition-carbonation process:
  - *equilibrium calculations* → suitable operating window (to be verified experimentally) & material selection for the process equipment (grinding media)
  - *coupling of thermodynamics with chemical kinetics* → process sizing & optimization

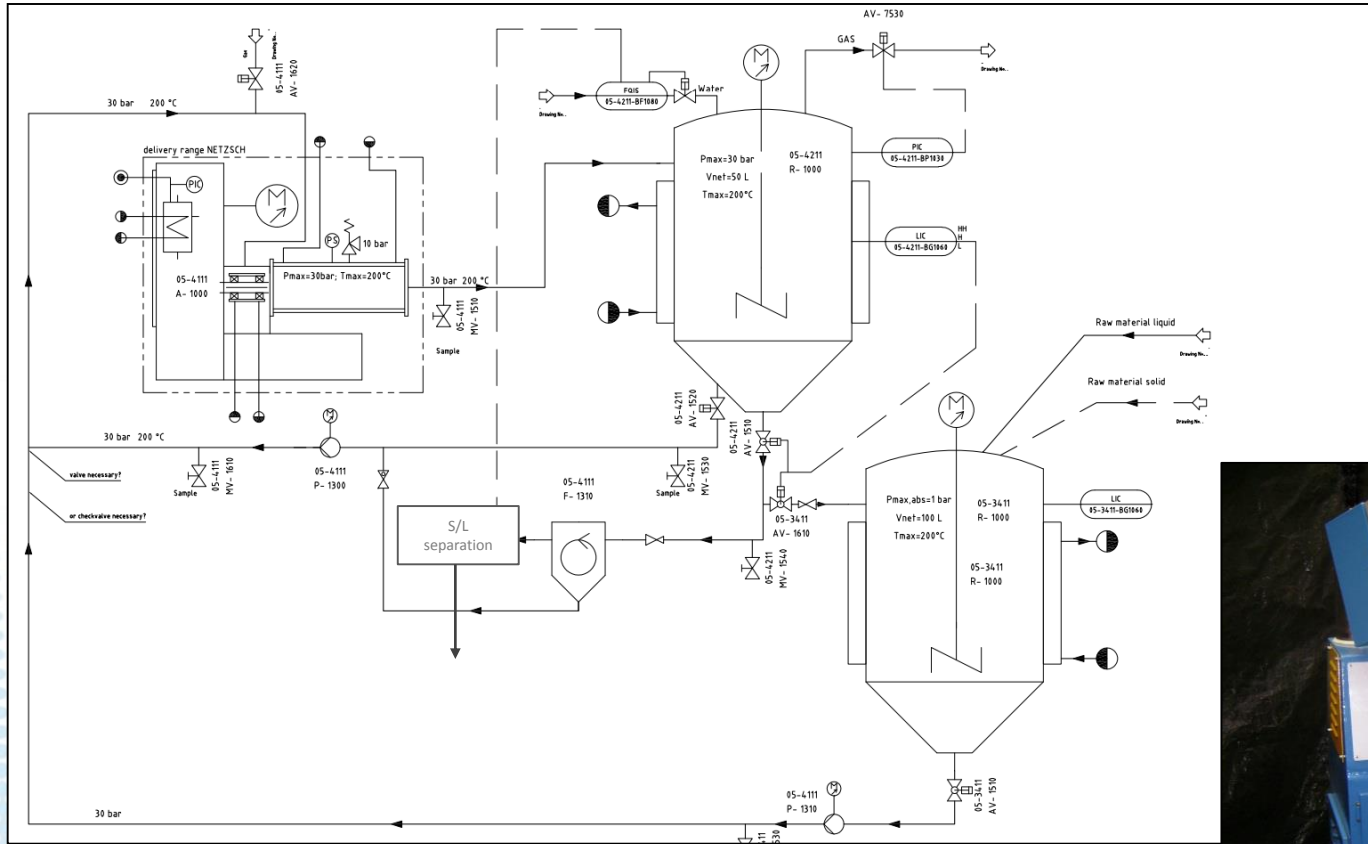
## SO WHAT'S NEXT ?

- **A continuous scalable demonstrator**
- **Beneficiation of carbonation products**





# Continuous bench-scale process



Attrition reactor ~4 L operating under T (max 200°C) & P (max 30 bar)



<http://www.isamill.com>

**➔ Bench-scale pilot reactor to be built in the coming months**



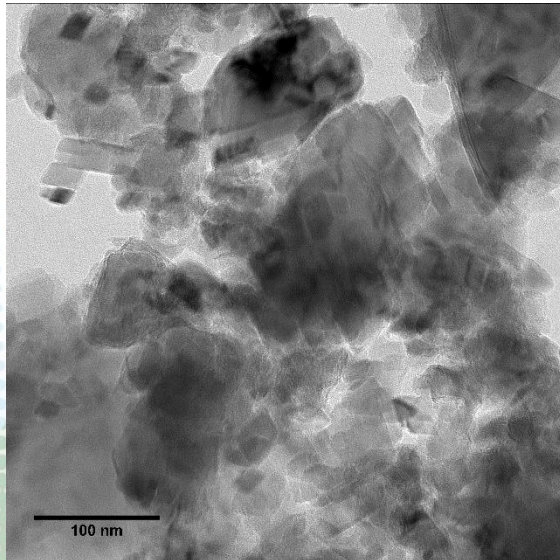


## Utilization of MC products as construction or filling materials

➤ could in theory absorb Gt of CO<sub>2</sub> (approximately 33 billion tons of concrete produced / year)

### Hybrid process products:

agglomerates of nano-sized particles of carbonates, silica & silicates



almost "as it is"

after heating

✓ Potentially highly reactive additives

Inert filler or pozzolana  
(mainly ultra-fine silica fraction  
+ MgCO<sub>3</sub>)  
CO<sub>2</sub> sequestered (& avoided)

Hydraulic binder  
(mainly Mg rich fraction  
+ SiO<sub>2</sub>)  
CO<sub>2</sub> avoided



**Goal:** valorization without solid separation & with minimum dewatering

- ✓ Virtuous CO<sub>2</sub> loop
- ✓ Reduction of natural resource depletion (no need for limestone)
- ✓ Reduction of heat demand (T<sub>calcination</sub> ↓)
- ✓ Local production of construction materials





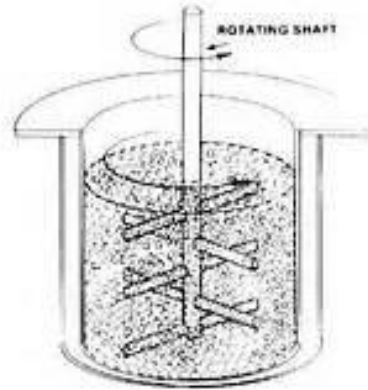


# New concept: CCUS combined with Enhanced Metal Recovery

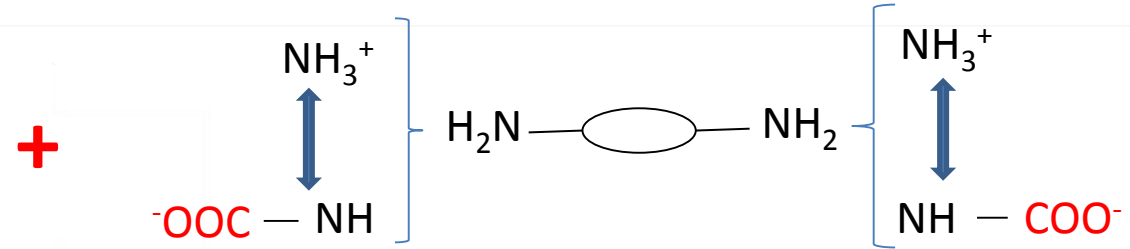
*Innovative chemistry under attrition environment*

## Attrition-carbonation process

CO<sub>2</sub>  
→  
→  
(Ni) slags



## Polyamines



efficient CO<sub>2</sub> capture (dilute flue gases)

+ catalysis of CO<sub>2</sub> mineralization

+ potential modulation of silica precipitation

+ selective metal recovery

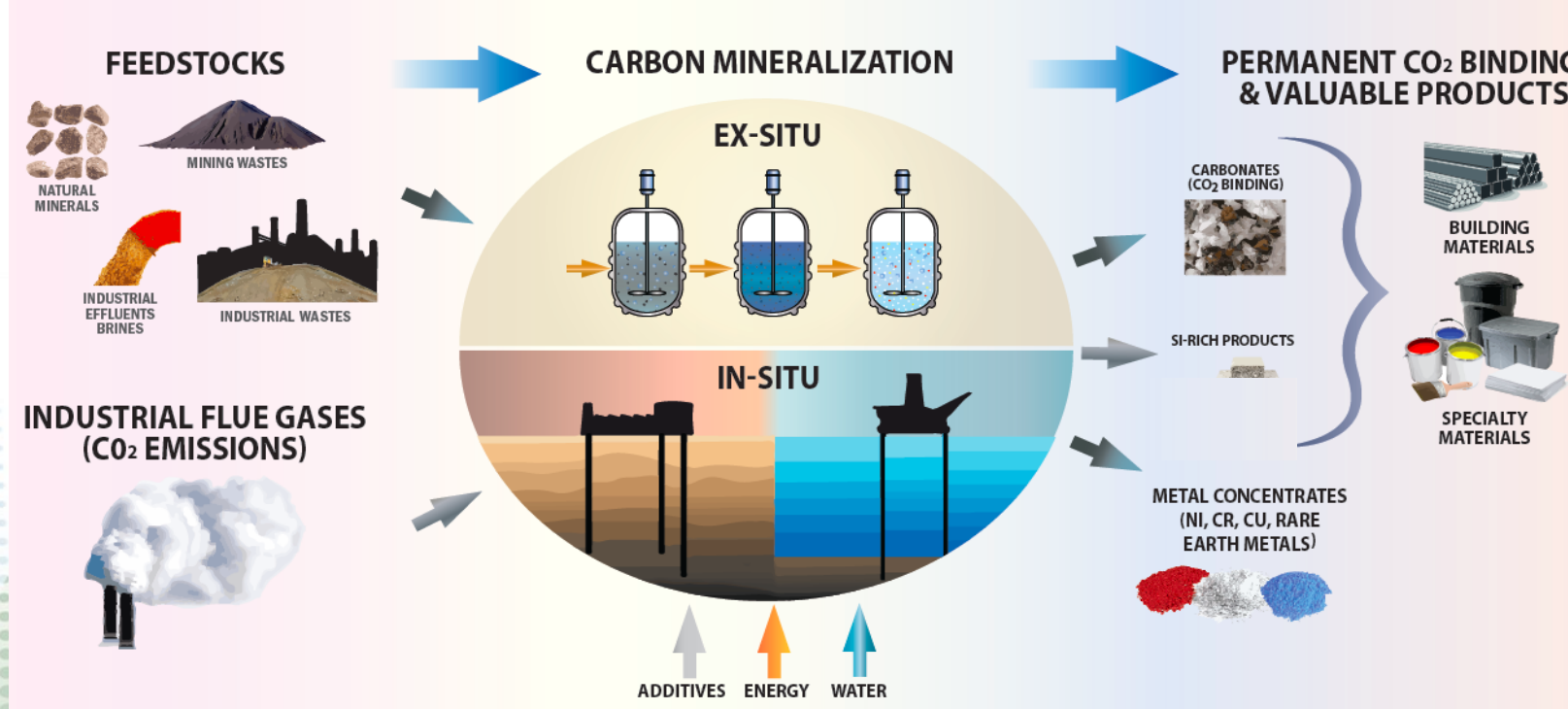
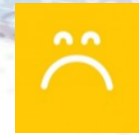




# Overall scheme for CO<sub>2</sub> mineralization

"Historically", CO<sub>2</sub> mineralization was compared to geological storage, in terms of storage cost and avoided CO<sub>2</sub>:

Limited development ...



Mission Innovation, CCUS\* Workshop, Houston, Sept. 2017; <http://mission-innovation.net/our-work/innovation-challenges>

In recent years, the mineralization of CO<sub>2</sub> systematically combines CO<sub>2</sub> storage and production of commercial goods, coupling environment and economy:

Booming development!





# Acknowledgements



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Caledonian Energy Agency



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F. Guyot, F. Ploquin, F. Juillot (**IMPMC Paris**)  
C. Petiot, L. Cassowitz, A. Boukary (**BioIS**)  
J. Leclaire & J. Septavaux (**ICBMS Villeurbanne**)  
for their valuable contribution



Thank you for your kind attention!





# The actual team



France Bailly, Director



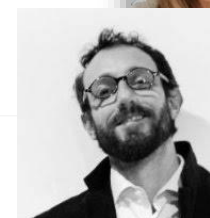
Pr. Florent Bourgeois



Dr. Carine Julcour



Dr. Laurent Cassayre



Pr. Julien Leclaire



Dr. Solène Touzé



Pr. Martin Cyr



