



September 22nd, 2021

Advanced Glass Up-Cycling by Alkali Activation

1222·2022
ANNI



UNIVERSITÀ
DEGLI STUDI
DI PADOVA

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²*Advanced Ceramics and Glasses group, Department of Industrial
Engineering, University of Padova, Italy*

 DIPARTIMENTO
DI INGEGNERIA
INDUSTRIALE



FunGlass

- **Binders for the building industry (e.g. Portland cement) generally having a significant environmental impact**
 - Gelation feasible at room temperature, but high temperature processing needed for the synthesis**
- **Interesting replacement offered by inorganic polymers, i.e. gels typically achieved by low temperature dissolution of alumino-silicate raw materials, in alkaline environment, followed by condensation reactions**
 - Demanding synthesis step still applied, for raw material preparation and/or definition of activating solution (e.g. alkali silicates)**

This paper aims at:

- **Confirming the potential of 'waste glass' as 'active' component of mixtures yielding IPs**
- **Defining products with different functionalities**

Unrecyclable glasses: a key example

Some glasses cannot be conveniently recycled

Strict requirements on chemical purity impose always 'virgin' raw materials

Data in wt%

Oxides	SL glass	BS glass
SiO ₂	71.6	72
Al ₂ O ₃	1.0	7
Na ₂ O	13.5	6
K ₂ O	0.4	2
MgO	3.9	
CaO	9.0	1
B ₂ O ₃		12
Fe ₂ O ₃	0.1	
TiO ₂		
SrO		
others	0.5	
L.O.I.		

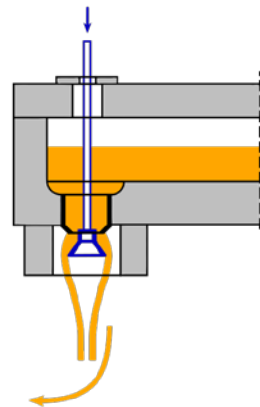
SL: Partially unrecyclable (highly contaminated fractions)

BS: Fully Unrecyclable

Images and glass cullet courtesy of Stevanato Group (Padova, Italy)



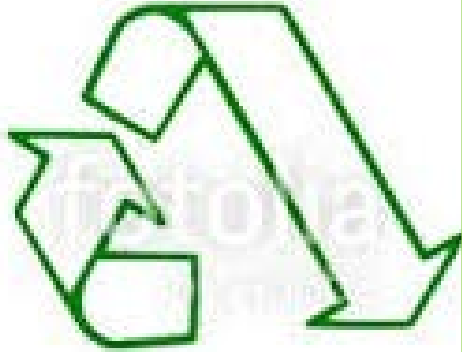
[<https://www.stevanatogroup.com/>]



True recycling unfeasible according to:

- Strict control of **chemical composition and quality**
- Industrial approach: glass pre-formed in form of tubes in one plant [Danner process] later transformed in another plant → **cullet hardly transported to primary manufacturer**

Valorization of discarded glass of any type
Attention to technologies not conditioned by the purity



Down-cycling
Savings from glass
as alternative raw
material (replacing
minerals)

**Traditional
ceramics (bricks,
stoneware tiles)
may include glass
in the formulation**



Up-cycling
Savings from
glass as
alternative raw
material
(replacing
minerals)

+ Added value

New glass-based engineering products

Key mission for the Advanced Ceramics & Glasses group, also in the framework of European Projects

GlaCERCo 2010-2014

CoACH 2014-2018

New-MINE 2016-2020

FunGLASS 2017-2024



Sustainable Up-Cycling: no-firing option

**No secondary thermal process, products exploited
for the replacement of energy-intensive
construction materials (e.g. conventional cement)**

Extensive glass reuse

- **FA (coal combustion) as 'usual' $\text{SiO}_2/\text{Al}_2\text{O}_3$ provider**
- **(Container) soda lime glass [SLG] providing BOTH SiO_2 AND alkali [Na_2O]**
- **Activation with NaOH solution [5-8-10 M]**

Mixtures designed to lead to:

$\text{SiO}_2/\text{Al}_2\text{O}_3 = 5 \rightarrow \text{FA/SLG} = 76/24$

$\text{SiO}_2/\text{Al}_2\text{O}_3 = 6 \rightarrow \text{FA/SLG} = 64/36$

$\text{SiO}_2/\text{Al}_2\text{O}_3 = 7 \rightarrow \text{FA/SLG} = 54/46$

Chemical composition of raw materials (wt%) determined by XRF.

wt%	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	MgO	Fe_2O_3	TiO_2
FA	54.36	24.84	0.83	3.03	2.56	2.06	8.28	1.07
SLG	70.5	3.2	12	1	10	2.3	0.42	0.07

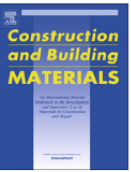
Construction and Building Materials 188 (2018) 1077–1084



Contents lists available at ScienceDirect

Construction and Building Materials

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



Extensive reuse of soda-lime waste glass in fly ash-based geopolymers

N. Toniolo^a, A. Rincón^b, J.A. Roether^c, P. Ercole^d, E. Bernardo^{b,*}, A.R. Boccaccini^{a,*}

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^dSasil S.p.a, Regione Dosso, 13862 Brusnengo, BI, Italy



Research carried out in the framework of European Community's H2020 MSCA-ITN 'CoACH-ETN', g.a. #642557
<http://www.coach-etn.eu/>



Idea: Avoiding alkali silicates

- **Useful in enhancing the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio**
- **Expensive**, since they imply a preliminary **melting stage (and dissolution \rightarrow 'water glass')**

Key points of Geopolymers

- Condensation of **alumino-silicate hydrated 'oligomers'** [poly-sialates]
- Oligomers from **alkali activation of alumino-silicate compounds** [dissolution in alkali or alkali-silicate aq. solutions]
- Stable structures [e.g. resisting boiling tests] for (amorphous or semi-crystalline) **zeolite-like structures**: [SiO_4] tetraedra mixed with [AlO_4] tetrahedra
- [AlO_4] tetrahedra stabilized by alkali ions

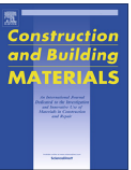
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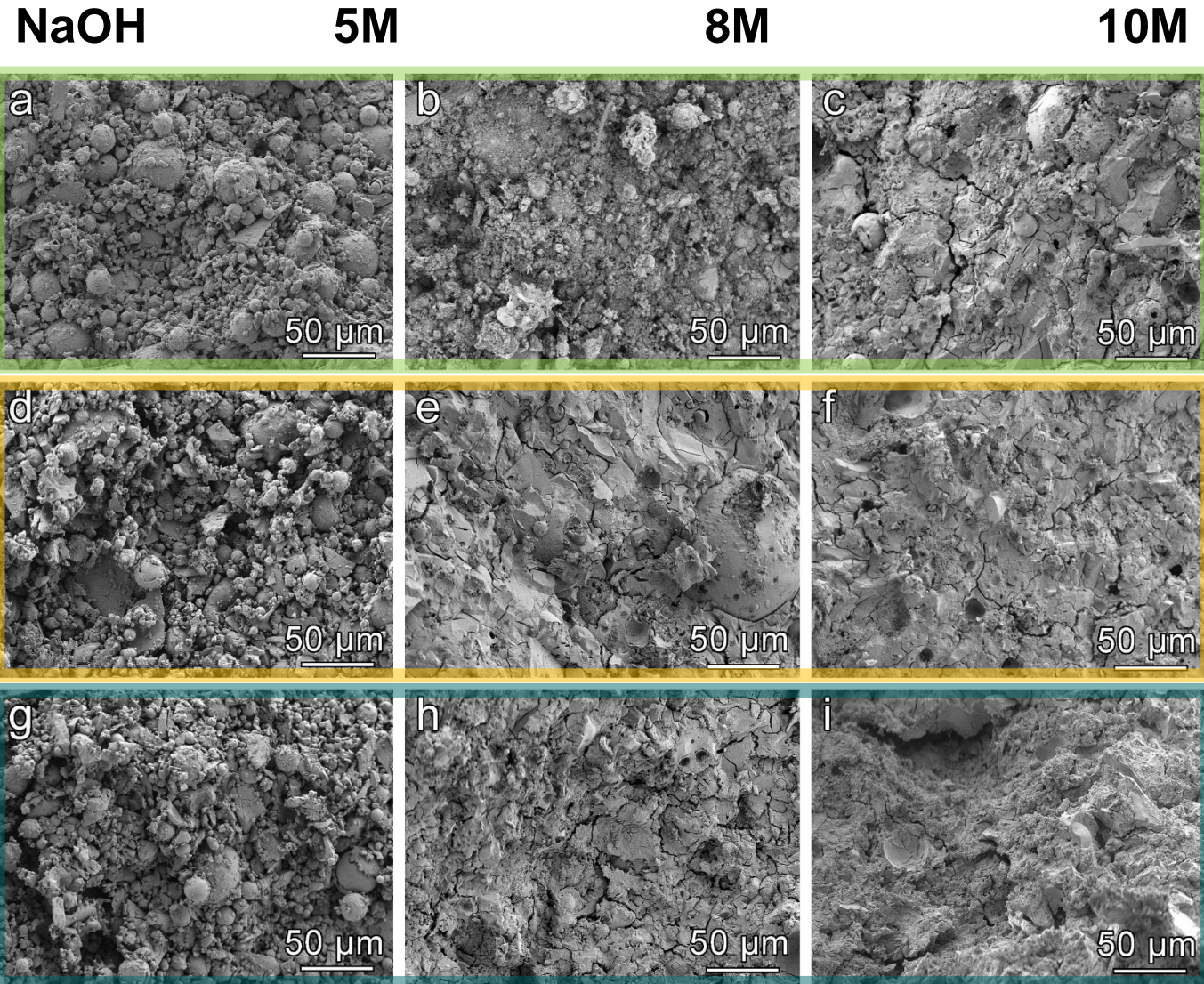
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$\text{SiO}_2/\text{Al}_2\text{O}_3 = 7 \rightarrow \text{FA/SLG} = 54/46$

- Fine powders (FA < 20 μm , SLG < 30 μm)
- 4h dissolution (solid/liquid=0.45), under mechanical stirring
 - Curing 48 h at 60 °C



Stabilization of heavy metals

Element (ppm)	As	Cd	Cr	Cu	Mo	Pb	Se	Zn
6S5M	0.174	0.007	0.0251	0.11	0.363	0.1	0.038	<0.203
6S8M	0.134	0.004	0.0101	0.115	0.143	0.078	0.048	<0.203
FA	<0.049	<0.002	0.467	0.028	0.898	<0.047	0.022	<0.2
SLG	<0.049	0.001	0.043	0.036	0.007	0.018	0.018	0.088
Inert material	0.5	0.04	0.5	2	0.5	0.5	0.1	4
Non-hazardous material	2	1	10	50	10	10	0.5	50

Mixtures designed to lead to:

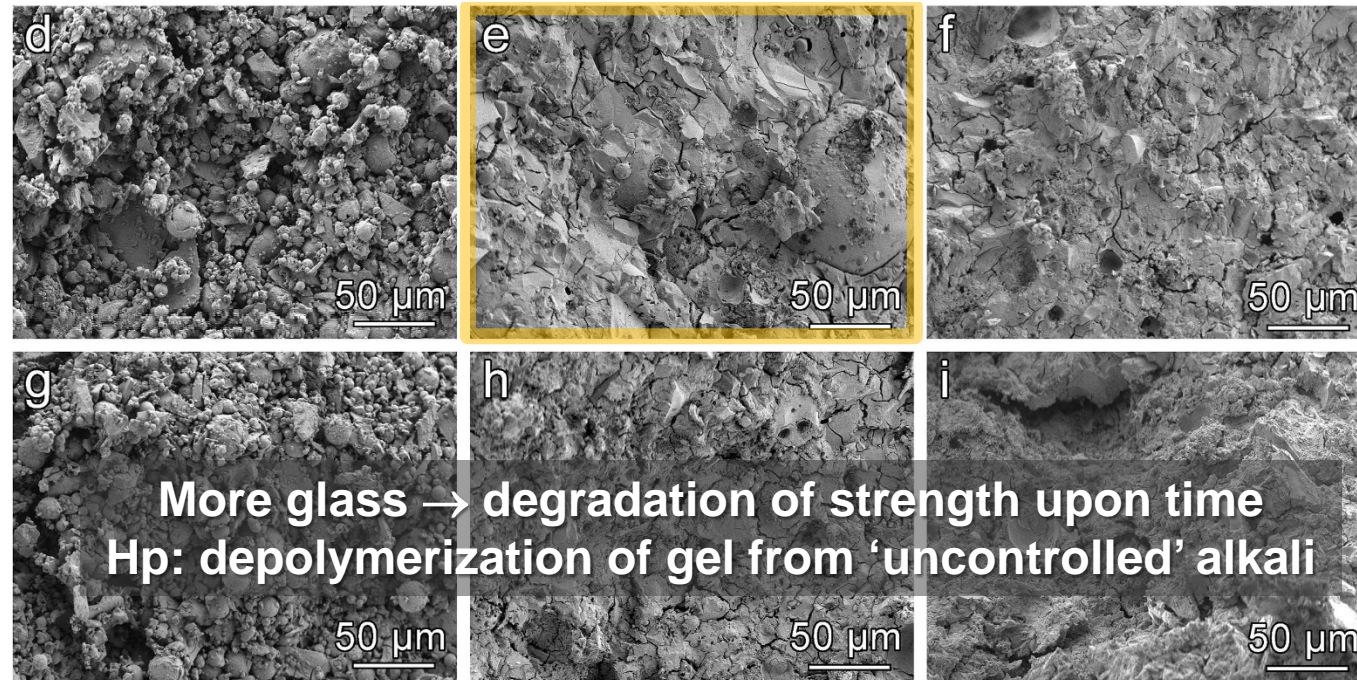
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Best: evidence of reaction, higher compaction (**density 1.93 ± 0.01 g/cm³, porosity 17 vol%**)

Stable compressive strength (>45 MPa, after 7 and 28 days)



Instead of

Reactive $\text{SiO}_2/\text{Al}_2\text{O}_3$ raw material + (Na_2O or $\text{Na}_2\text{O}/\text{SiO}_2$) activator \rightarrow GP

‘Alternative approach’ to geopolymers:

Reactive SiO_2 raw material + ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$) activator \rightarrow GP

E.g. silica fume, rice husk ash, microsilica [=agricultural or industrial waste]

Fundamental papers by *K.J. MacKenzie and collaborators [Victoria University of Wellington, NZ]*

G. Gluth & C. Jäger and collaborators [BAM, Berlin, Germany]

e.g. Brew et al., *J. Mat . Sci.* **42**, 3990–93 (2007)
Greiser et al., *RSC Advances* **8**(70), 40164-71 (2018)

This paper:

- **Fine soda-lime glass as SiO_2 precursor** \rightarrow Attention: **SLG actually providing also Na_2O !**
- Activation by means of **aqueous solution of commercial NaAlO_2**

Starting solution: 45 wt % NaAlO_2 in distilled water;
slow mixing at room temperature for 30 min

Starting solution: **45 wt % NaAlO₂** (Sigma Aldrich, Gillingham, UK);
slow mixing at room temperature for 30 min

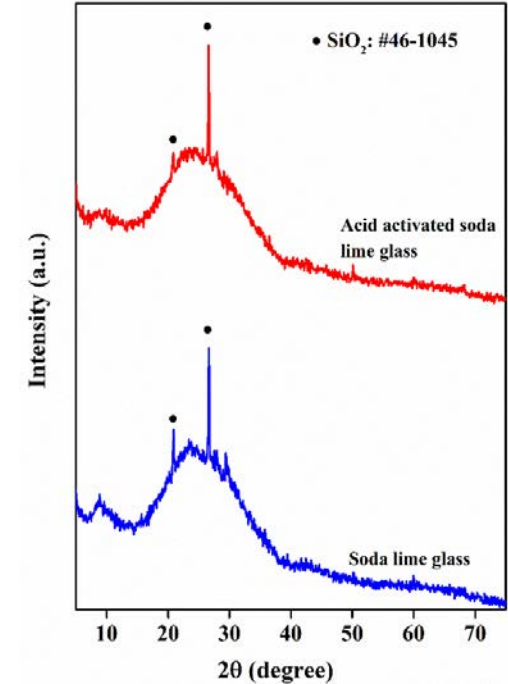
Route #1

- Fine powder of SLG (30µm in size) cast in NaAlO₂ solution, in a proportion 50/50 wt %
- Mixing for 3h, under the low speed mechanical stirring (300 rpm)
- Casting in PS moulds and curing at 75 °C, 7 days

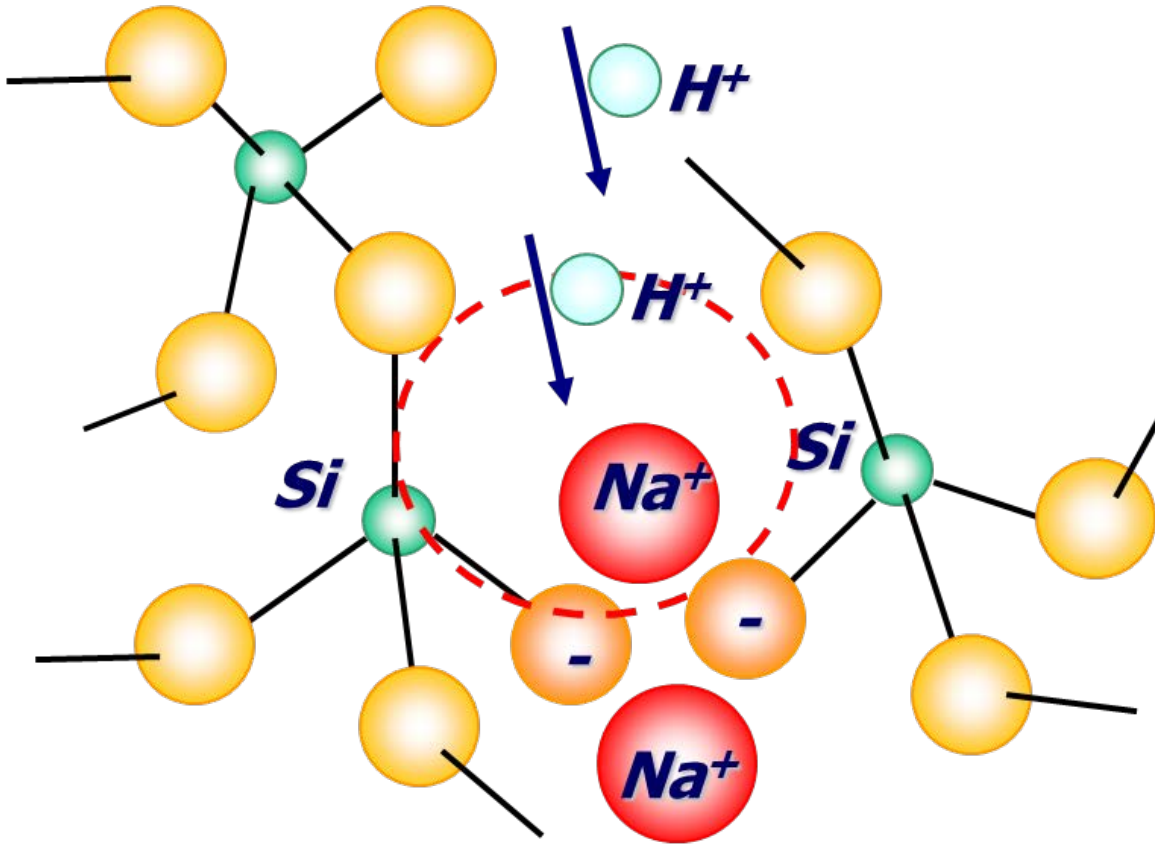
Route #2: preliminary acid leaching

- Fine powder of SLG (30µm in size) cast in distilled water; pH = 5 by addition of concentrated HCl solution
- 3h leaching (pH monitored and kept ~ 5 by periodical dropwise addition of HCl solution)
- Washing and centrifugation in distilled water (6 cycles) + drying at 40 °C, overnight
- Fine powder of treated SLG cast in NaAlO₂ solution, in a proportion 50/50 wt %
- Mixing for 3h, under the low speed mechanical stirring (300 rpm)
- Casting in PS moulds and curing at 75 °C, 7 days

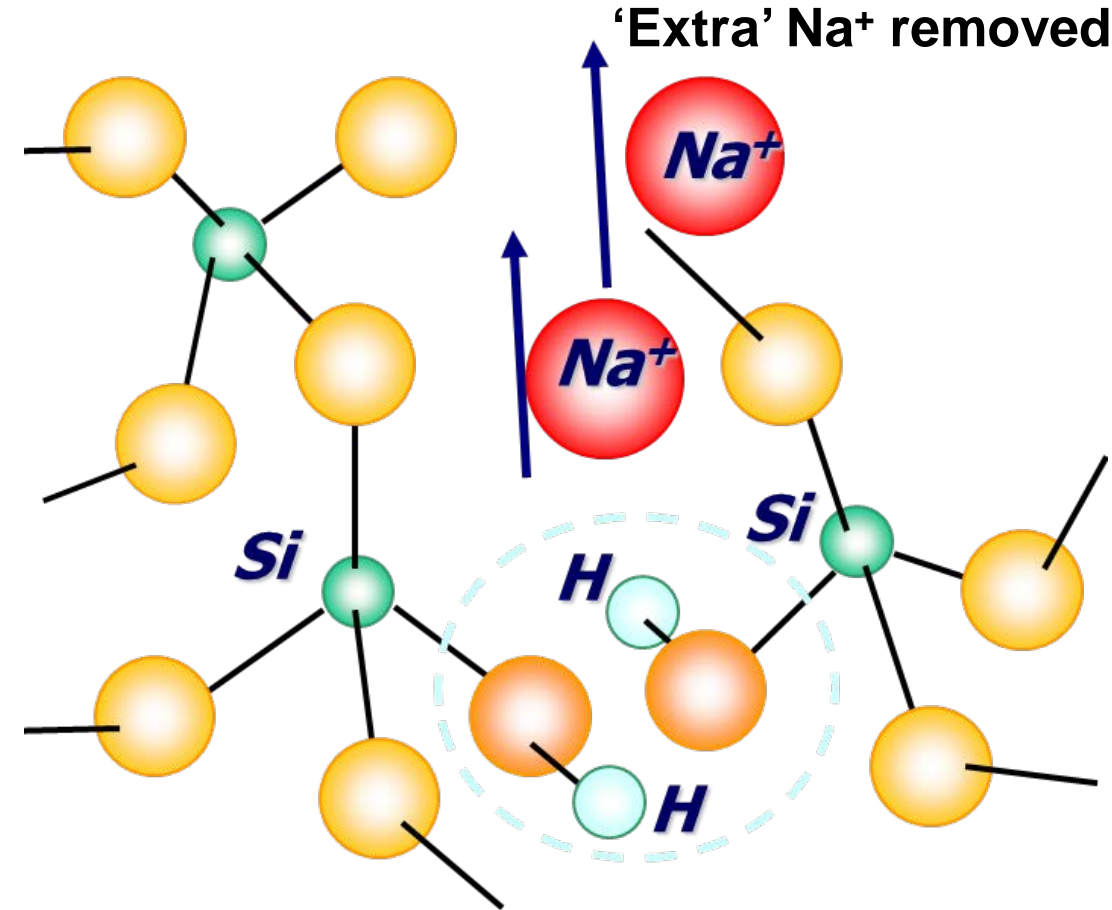
Overall
NaAlO₂/SLG
= 0.31



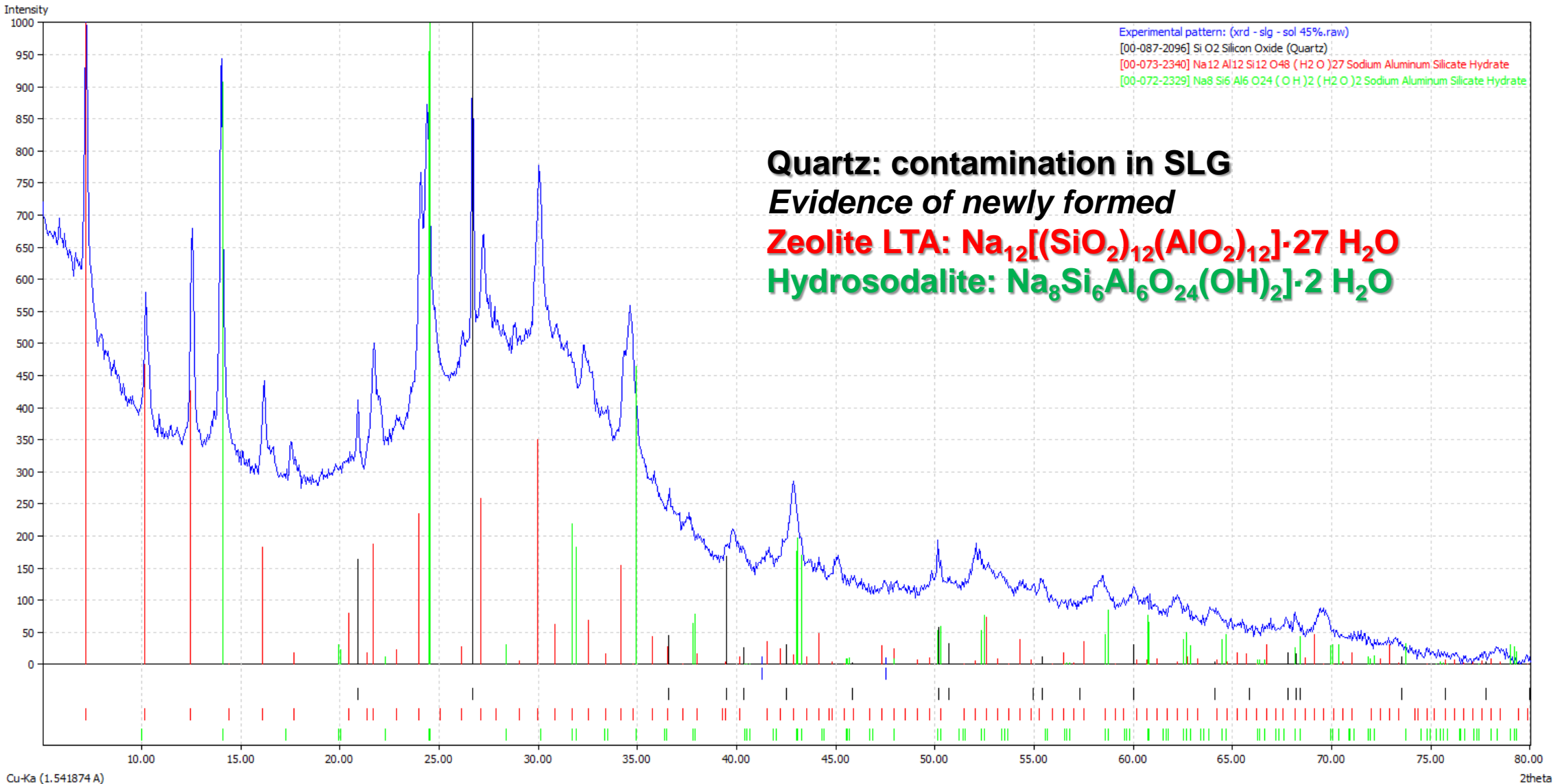
Preliminary test: survival in boiling water [30 min; 4g in 50 cl]→No disintegration, no dissolution



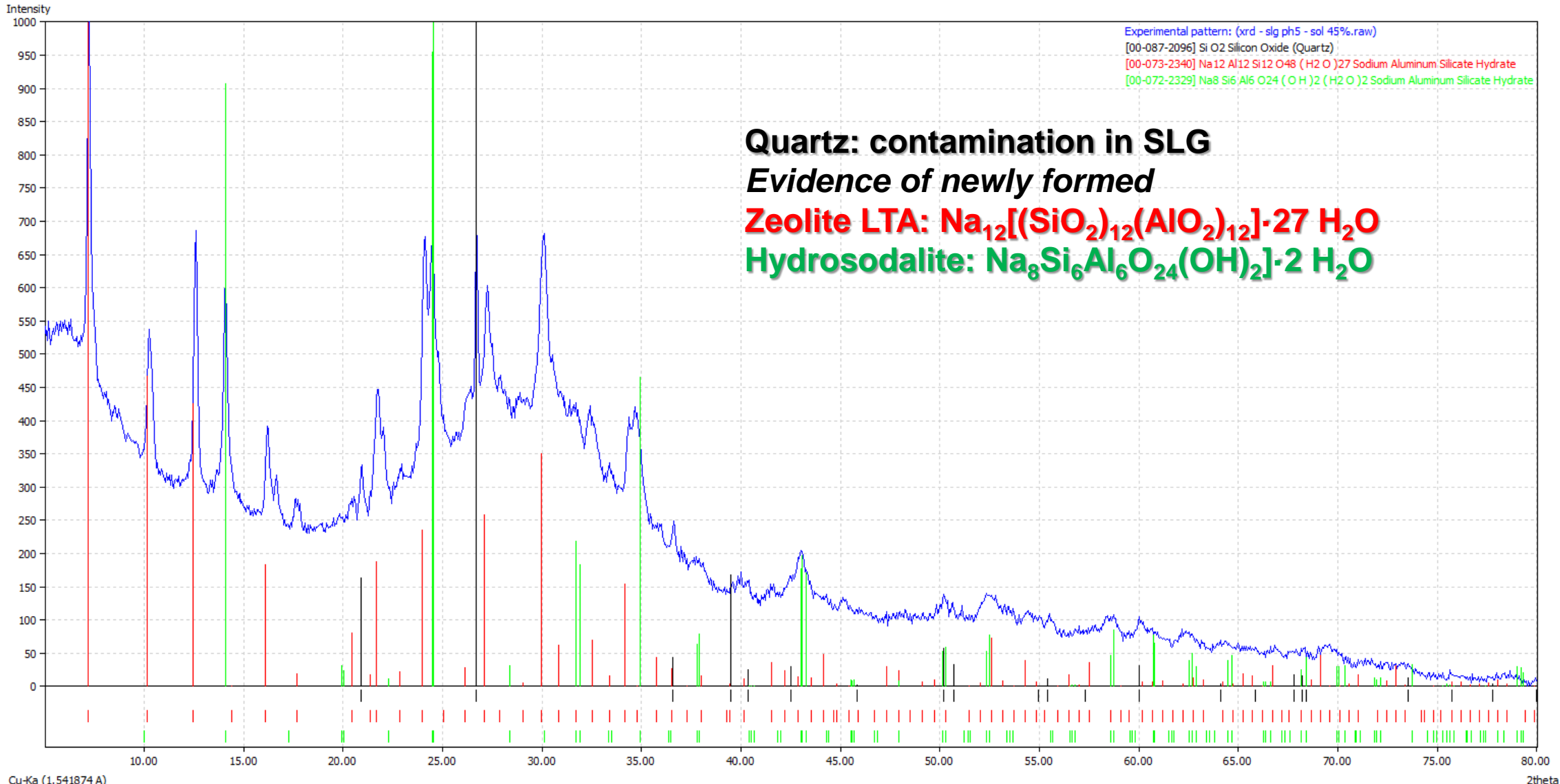
Acid leaching: protons replacing Na^+
Additional treatment, but it may be applied to recover metals (e.g. lamps, PV panels)

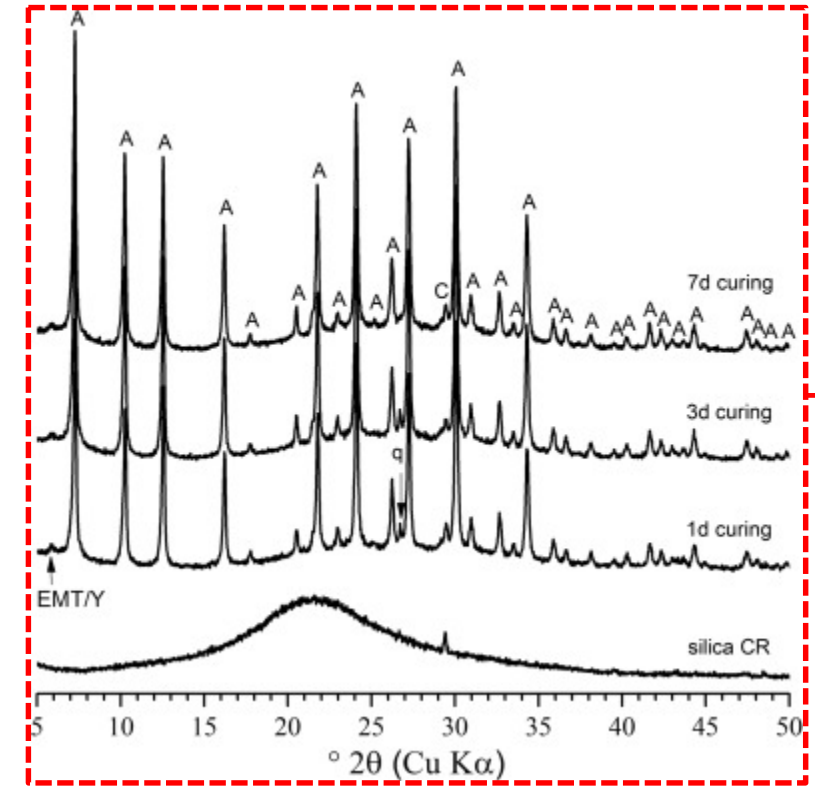
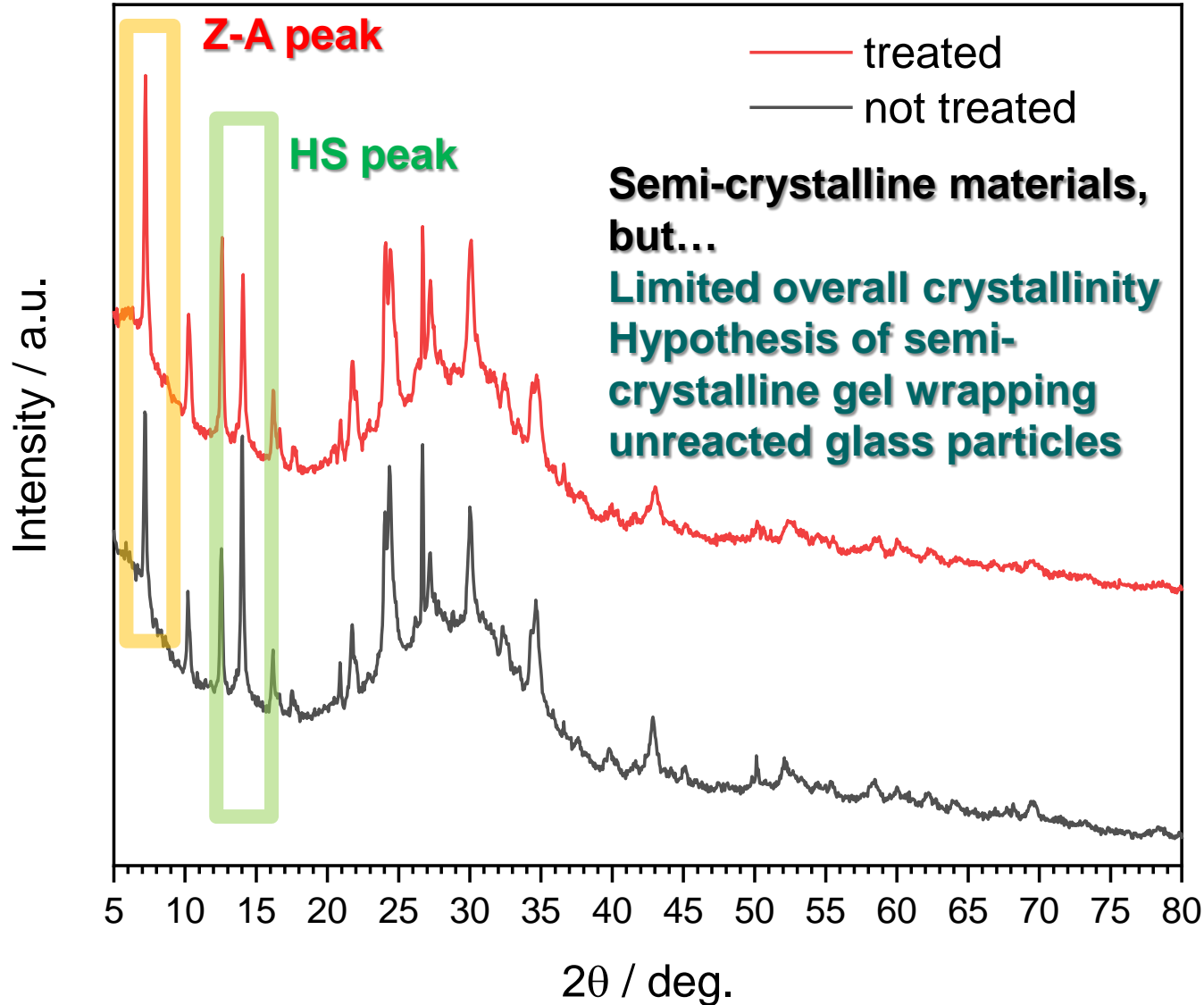


Hydrated silica expected to react with the activating solution (gelation with Na^+ just from solution)

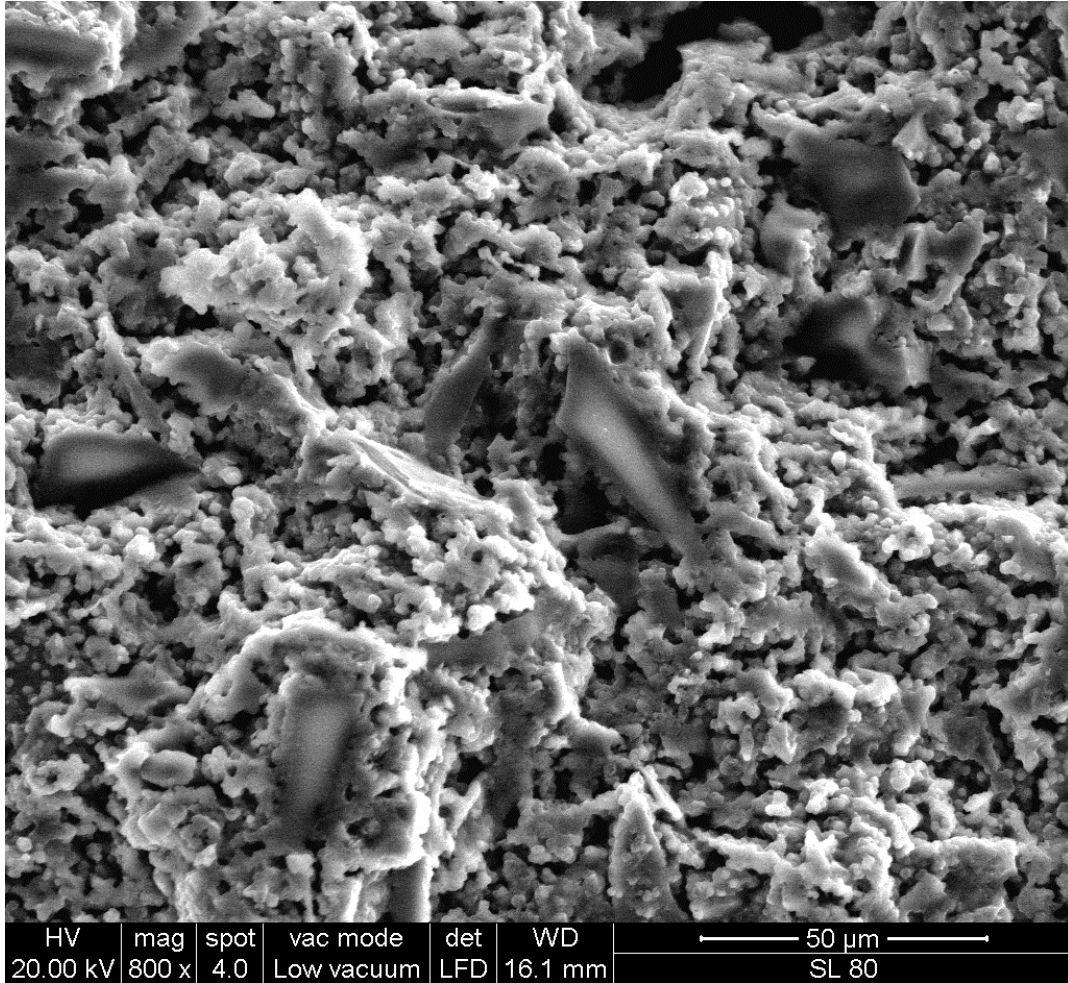


Acid leaching: semi-crystalline material again, but different
Hydrosodalite (reduced) + Zeolite LTA (enhanced)





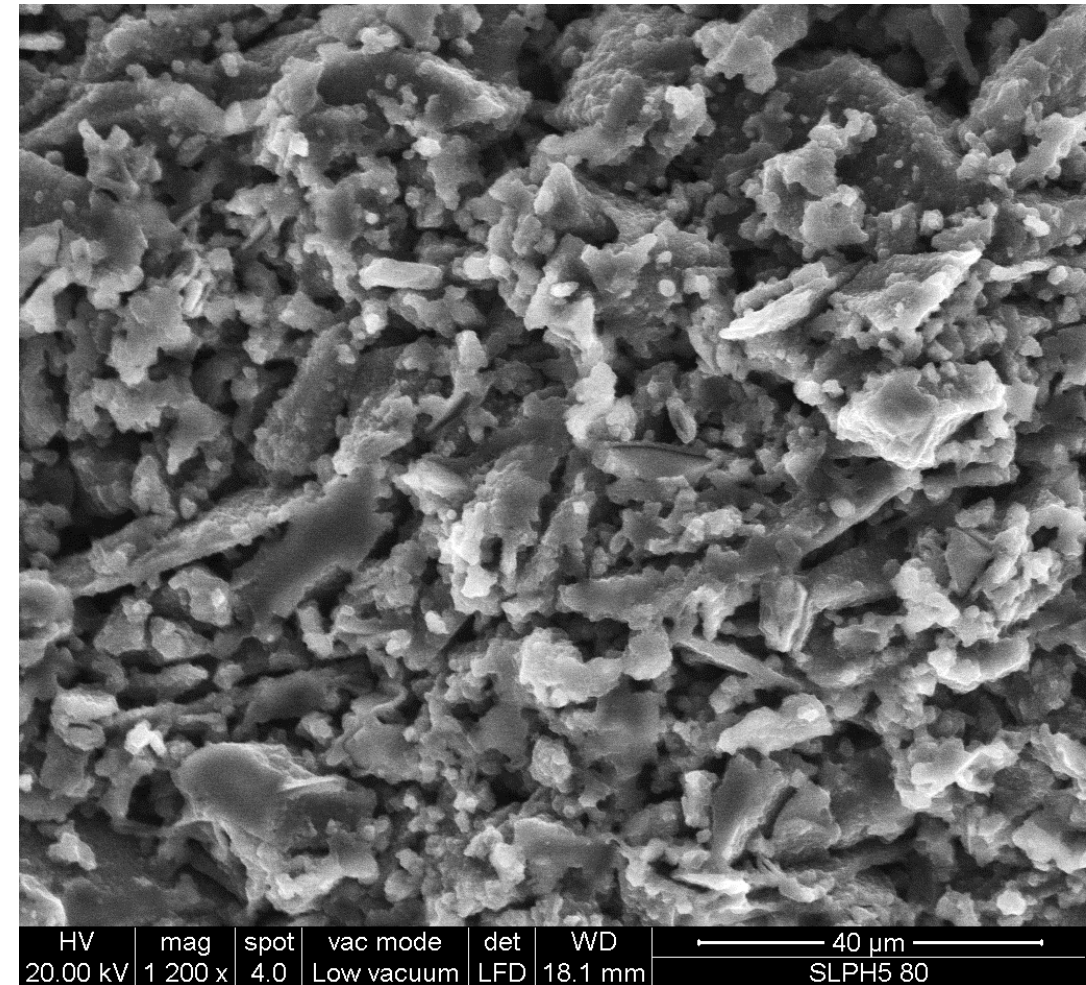
Interesting comparison with '**Geopolymer-zeolite composites**' from NaAlO_2 and silica waste (from chlorosilane production)
Greiser et al. Ceram. Int. 43(2), 2202-8 (2017)

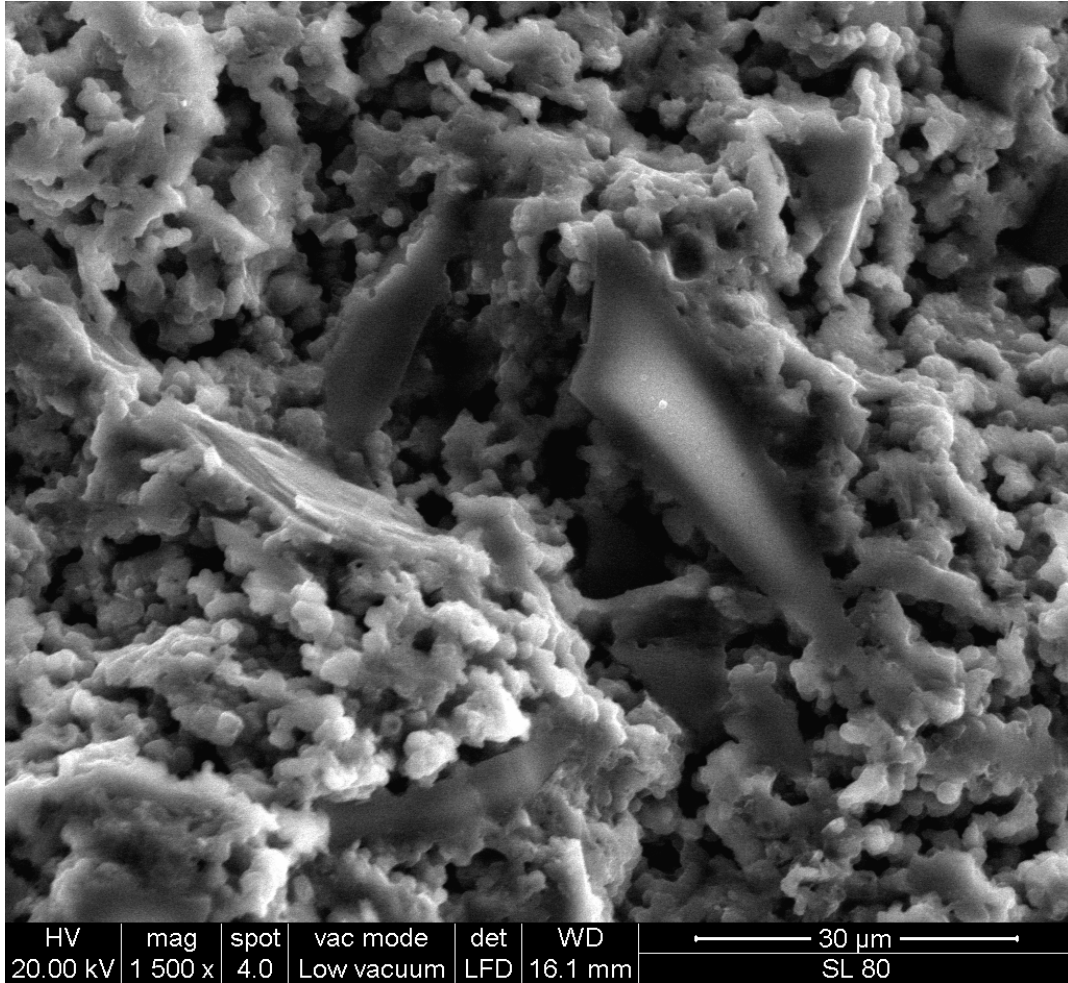


SLG with no treatment: well recognizable former glass granules

Binding phase 'attached' to glass particles

SLG after leaching: binding phase integrated at the surface



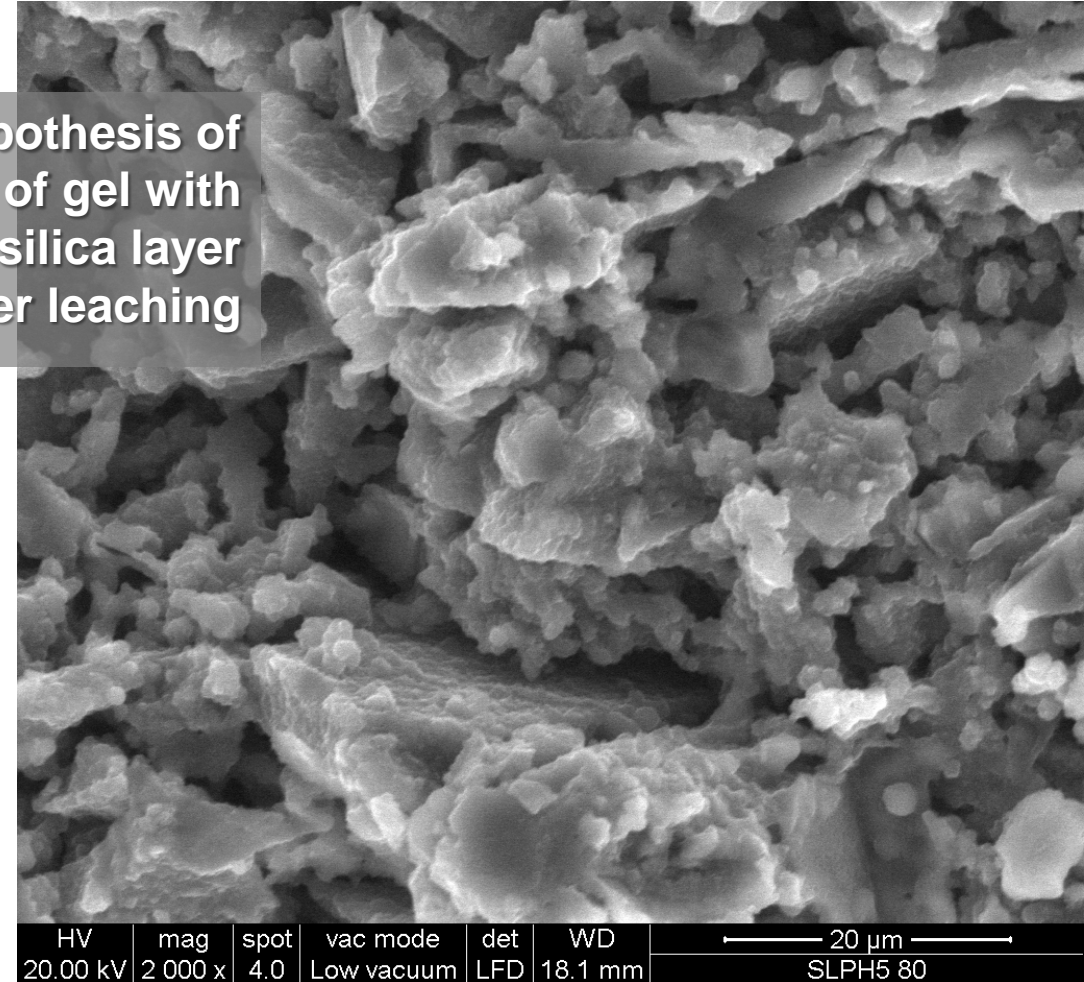


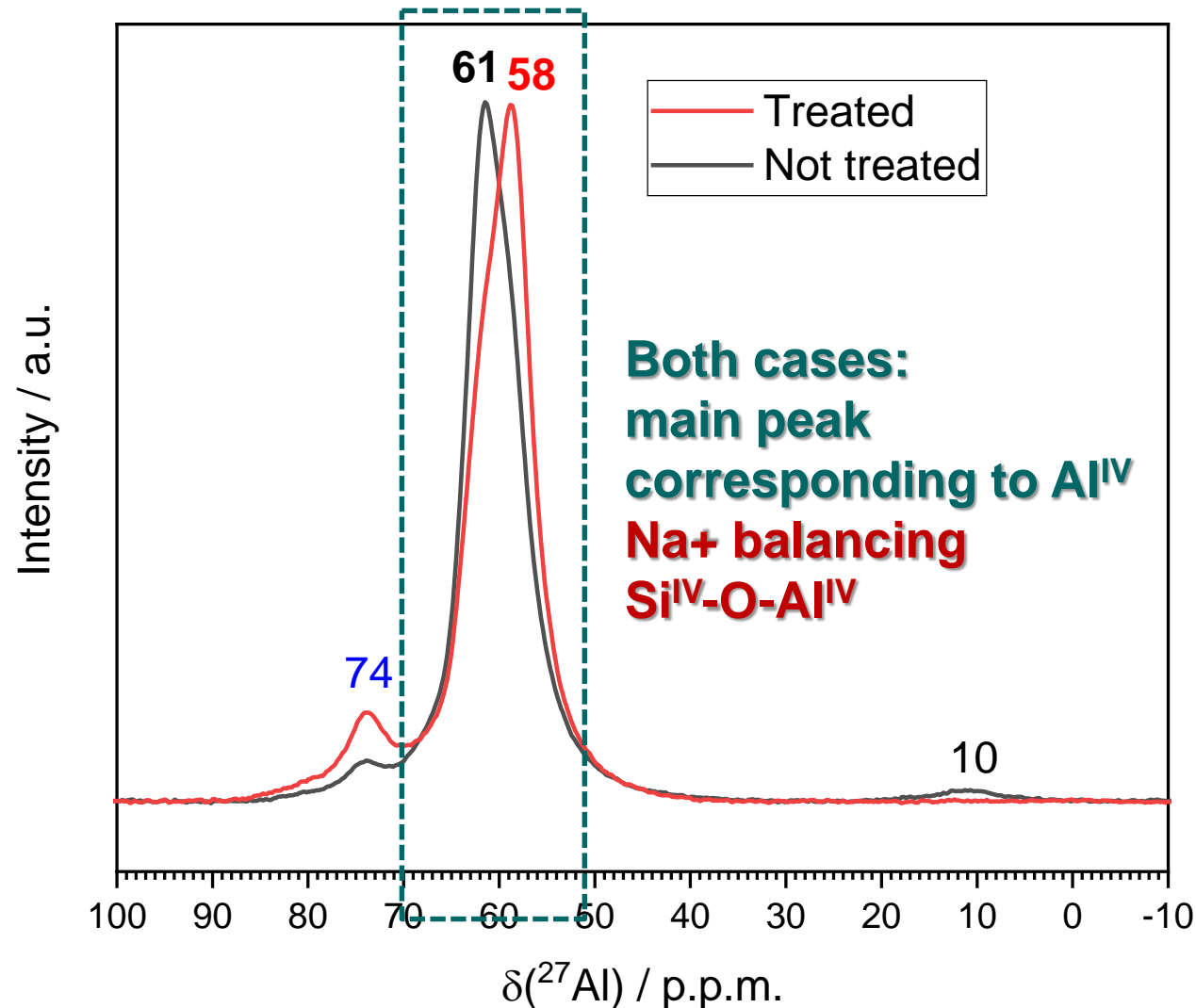
SLG with no treatment: well recognizable former glass granules

Binding phase 'attached' to glass particles

SLG after leaching: binding phase integrated at the surface: **texturing**

Hypothesis of integration of gel with hydrated silica layer available after leaching





- **No signal attributable to unreacted NaAlO_2 ($\delta \sim 80$ ppm)**
- **Some signal consistent with Al^{VI} in materials from not treated SLG (AlO_6 from minor amounts of $\text{Al}(\text{OH})_3$)**

Brew et al., J. Mat. Sci. **42**, 3990–93 (2007)

- **Some signals consistent with Al^{V} in both materials ($\delta \sim 74$ ppm)**

Walkley et al., J. Phys. Chem. C **122**, 5673–85 (2018)

More interestingly, for Al^{IV} :

Exchange of main peak and shoulder

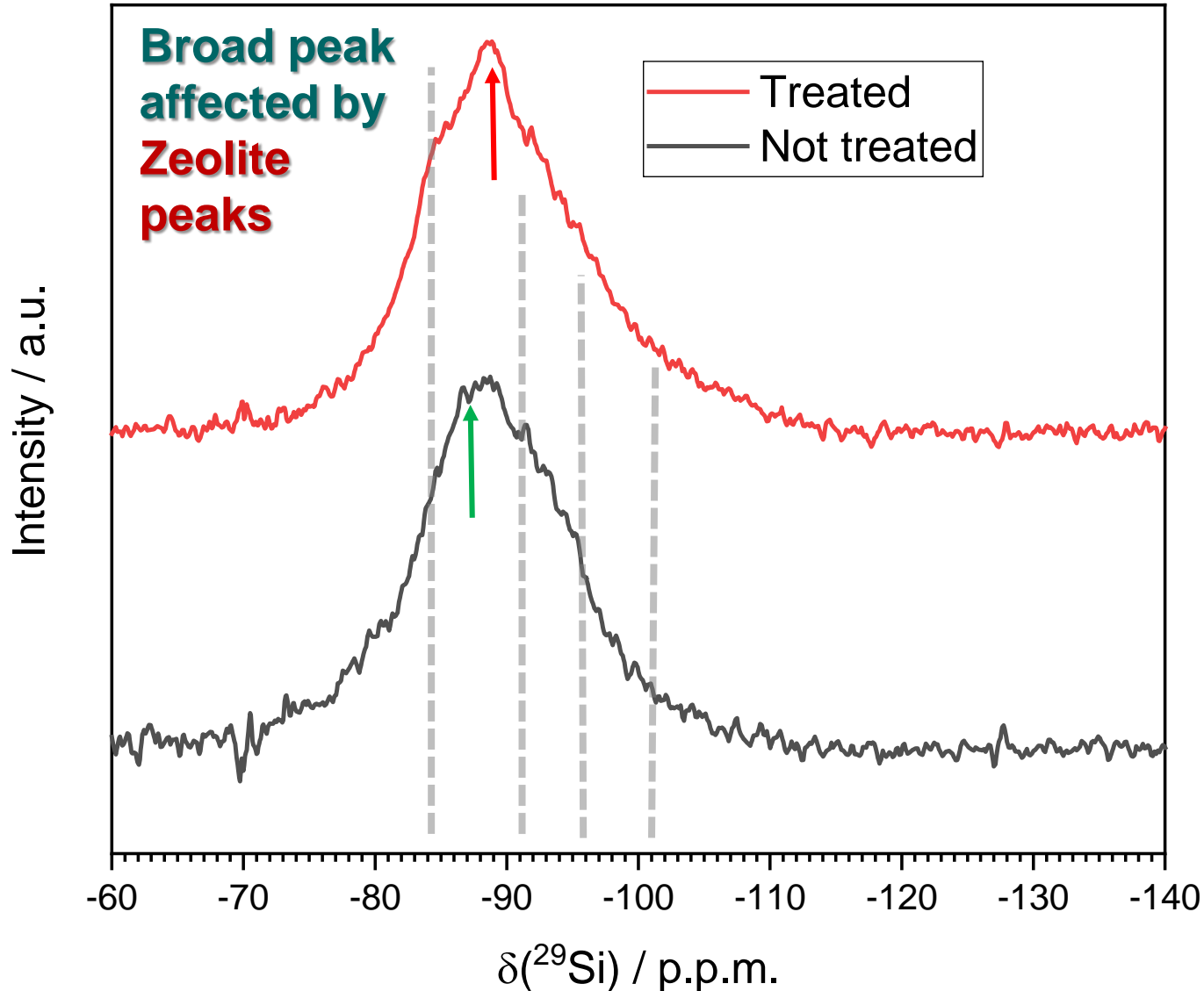
Not treated $\delta \sim 61$ ppm [shoulder at ~ 58]

less ordered AlO_4 units

Walkley et al., J. Phys. Chem. C **122**, 5673–85 (2018)

Treated $\delta \sim 58$ ppm [shoulder at ~ 61]

Greiser et al., RSC Advances **8**(70), 40164–71 (2018)



Interesting match with assignments from the literature

Greiser et al., RSC Advances 8(70), 40164-71 (2018)

$\delta \sim -86$ ppm (not treated):
hydrosodalite

$\delta \sim -89$ ppm (treated): **zeolite LTA**

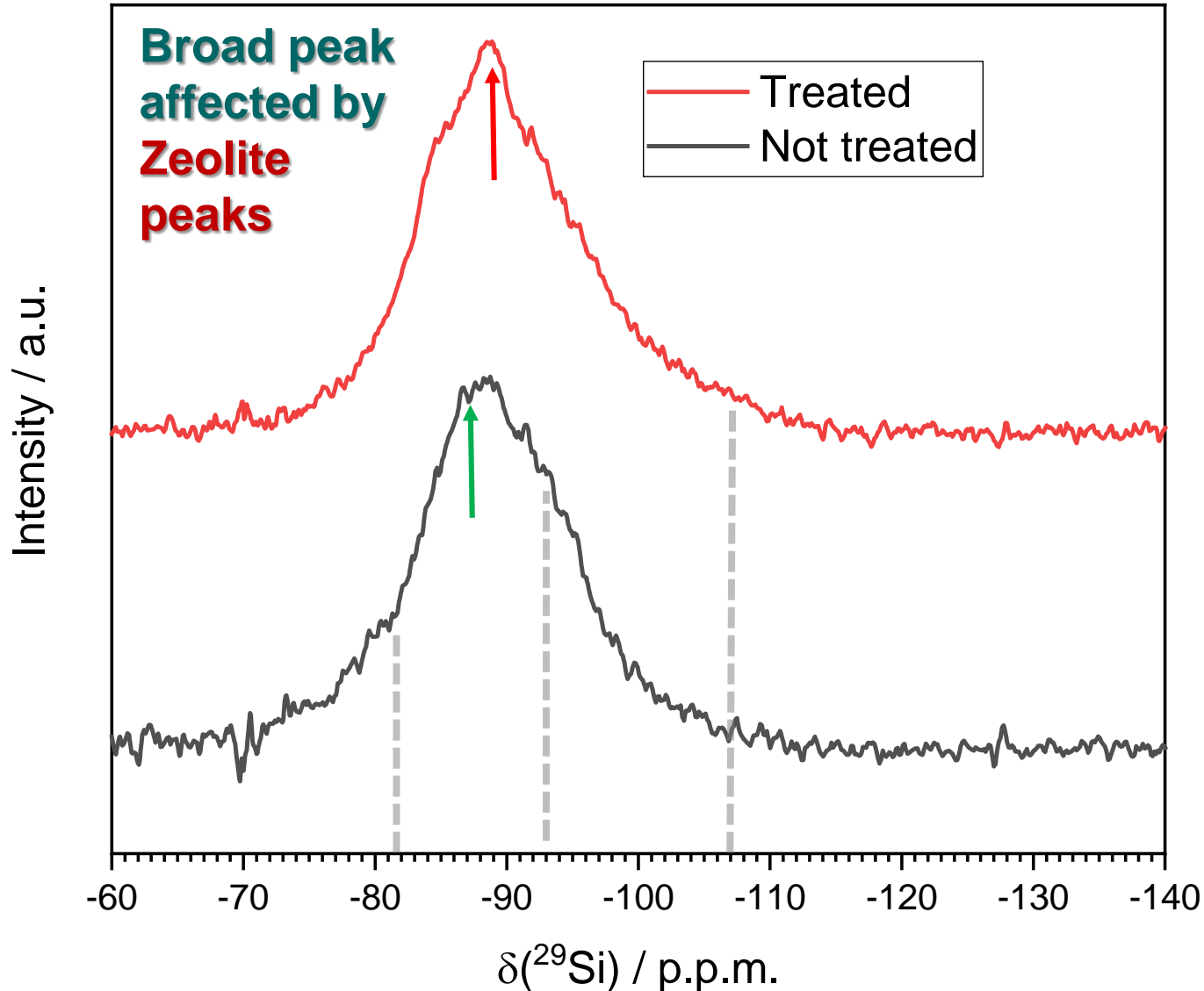
Signals consistent with Q^4 species in alumino-silicate gel

$\delta \sim -84$ ppm **Q^4 (4Al)**
[more for T]

$\delta \sim -91$ ppm **Q^4 (3Al)**

$\delta \sim -96$ ppm **Q^4 (2Al)**
[more for NT]

$\delta \sim -102$ ppm **Q^4 (1Al)**



Interesting match with assignments from the literature

Signals consistent with SiO_4 species in the presence of Na^+ and Ca^{2+} ions

Jones et al., J. Non-Cryst. Sol. 293-295, 87-92 (2001)

$\delta \sim -82 \text{ ppm}$ Q^2
[more for NT]

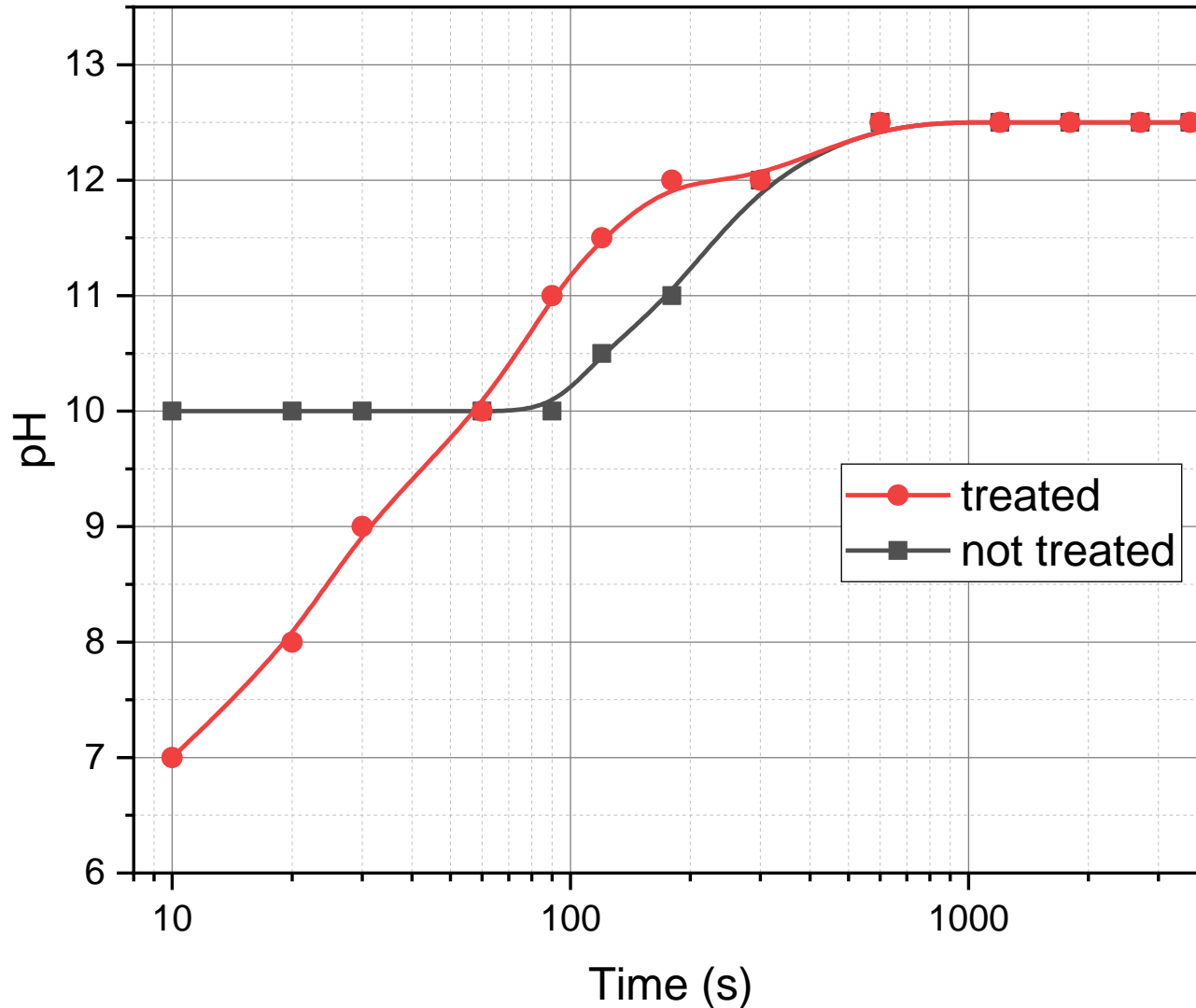
$\delta \sim -93 \text{ ppm}$ Q^3

$\delta \sim -107 \text{ ppm}$ Q^4
[more for T]

Hypothesis of more polymerized gel with acid pre-treatment

Alkali mobility test

Crushed gels left in distilled water
Sudden alkalization with not treated glass



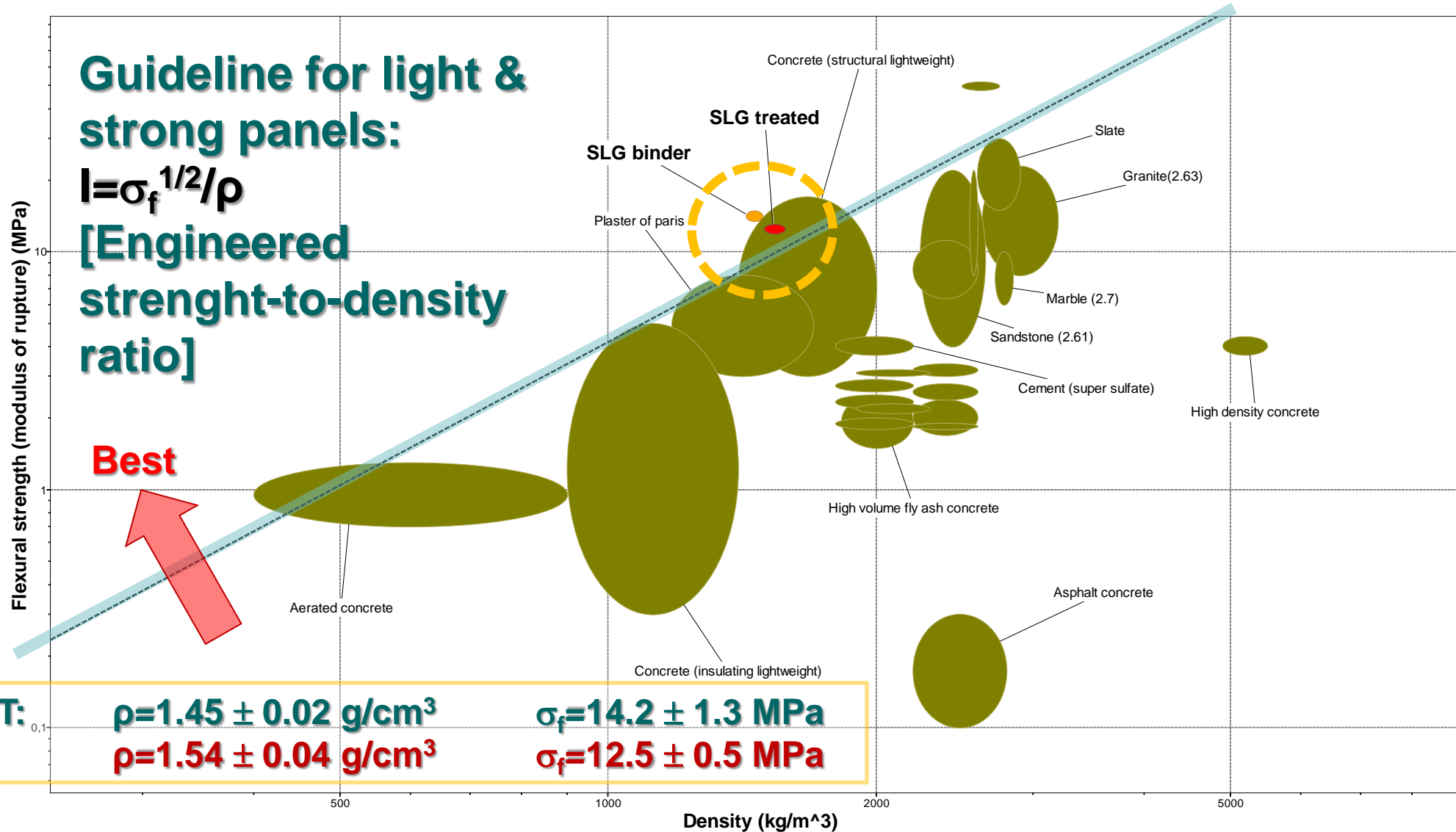
Gels crushed <2 mm

Cast in distilled water (35% solid loading)

Room temperature

Different stabilization of sodium ions

Effect detected also by simple hand contact



NT: $\rho = 1.45 \pm 0.02 \text{ g/cm}^3$ $\sigma_f = 14.2 \pm 1.3 \text{ MPa}$
T: $\rho = 1.54 \pm 0.04 \text{ g/cm}^3$ $\sigma_f = 12.5 \pm 0.5 \text{ MPa}$

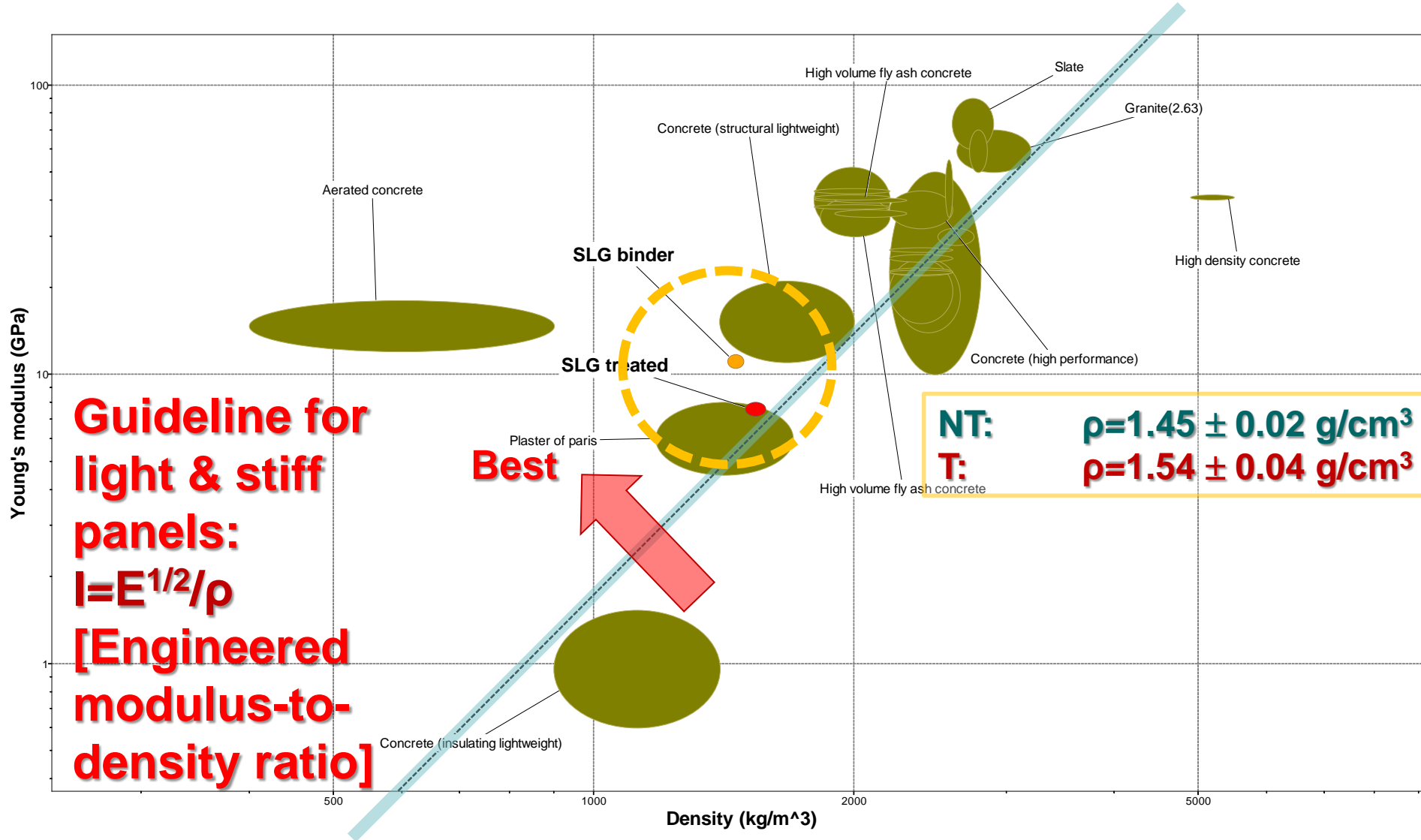
New cementitious materials comparing well with commercial ones in terms of strength

Graph and ref. data from CES (Cambridge Engineering Selector) EduPack 2019 [GrantaDesign, Cambridge, UK]

Basic mechanical characterization

Glass-based gels comparable with concrete products

Elastic modulus



**Guideline for
light & stiff
panels:
 $I = E^{1/2} / \rho$
[Engineered
modulus-to-
density ratio]**

Best

**NT:
T:**

**$\rho = 1.45 \pm 0.02 \text{ g/cm}^3$
 $\rho = 1.54 \pm 0.04 \text{ g/cm}^3$**

**$E = 11.1 \pm 0.6 \text{ GPa}$
 $E = 7.56 \pm 0.4 \text{ GPa}$**

**New
cementitious
materials
comparing well
with
commercial
ones in terms of
stiffness**

**Graph and ref. data
from CES
(Cambridge
Engineering
Selector) EduPack
2019 [GrantaDesign,
Cambridge, UK]**

Extension: dilution with coarse glass

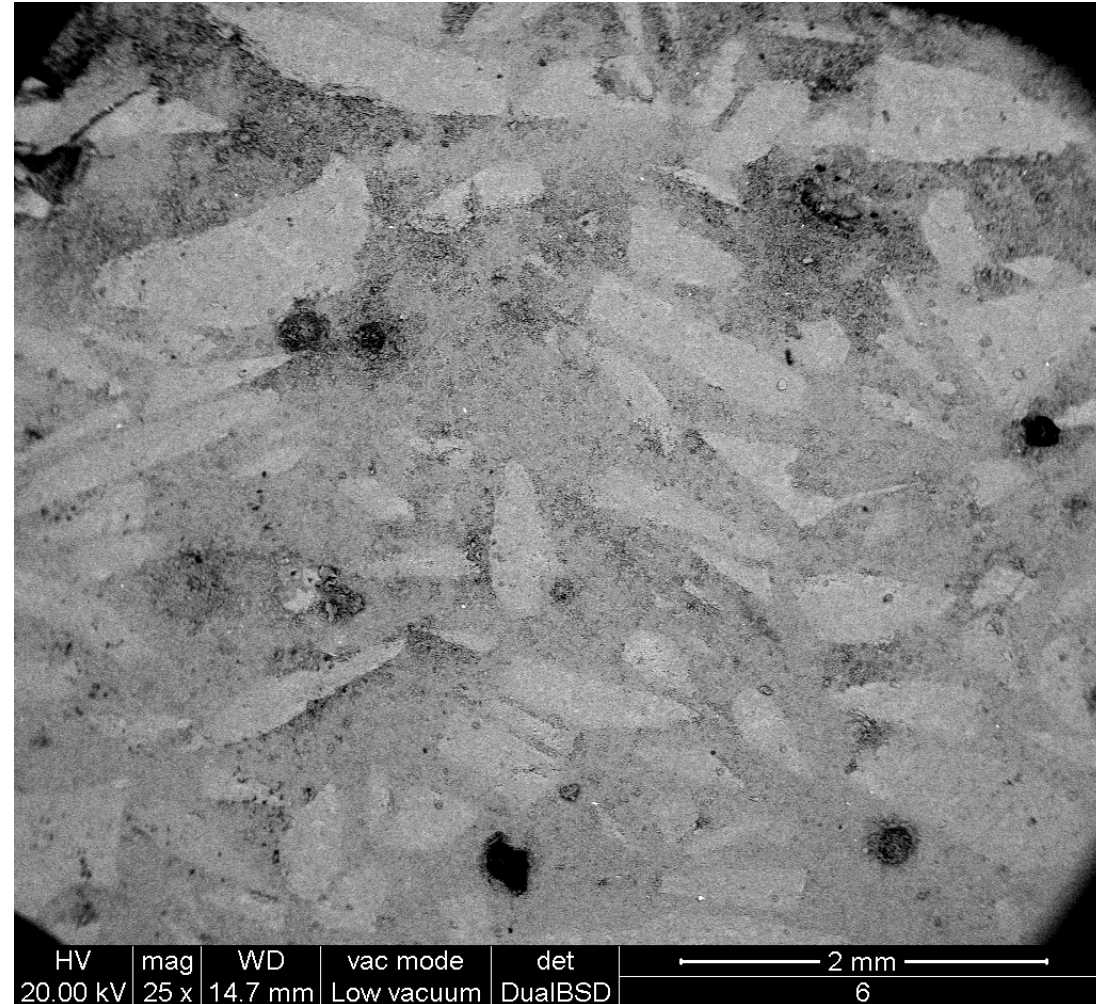
Glass-based concrete: enhancing sustainability
50wt% coarse glass added

Coarse glass fragments (crushed and sieved in the interval: 300-1400 μm) added as filler

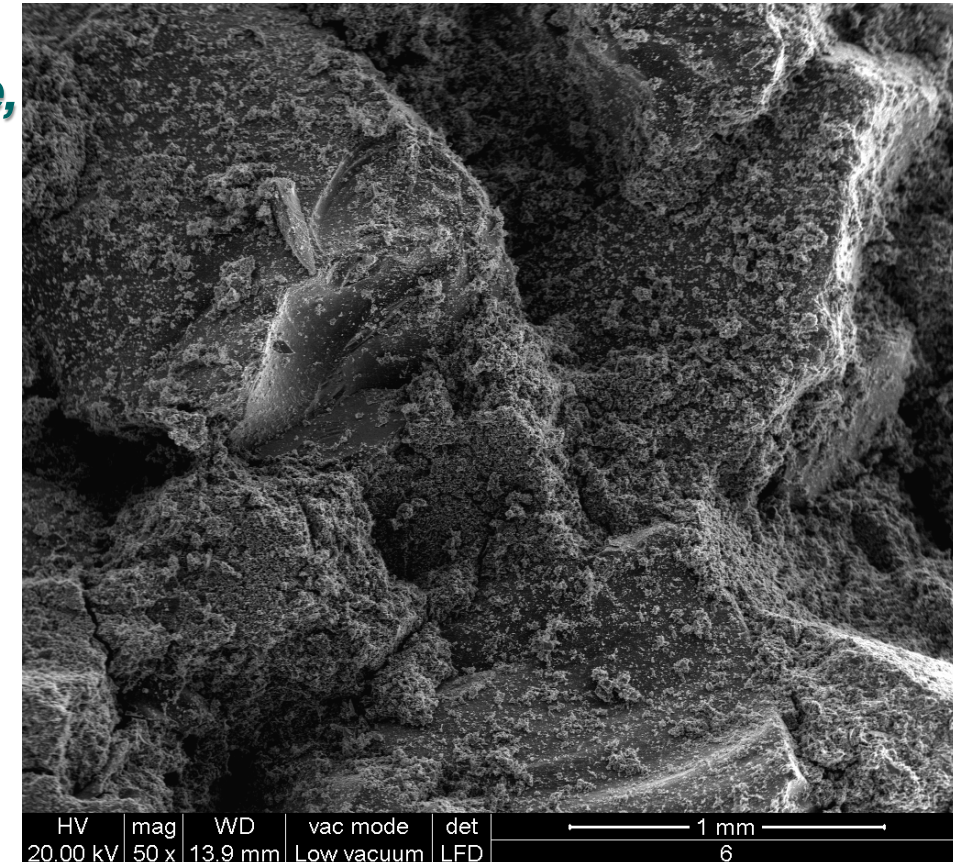
Binder from Acid Treated glass, fracture surface: coarse fragments coated by gel

Crack passing at the interface, but mostly remaining in the gel

Overall
NaAlO₂/SLG
passing from
0.31 to 0.14
[0.31 g NaAlO₂/
1g fine
SLG/1.31 g
coarse SLG]

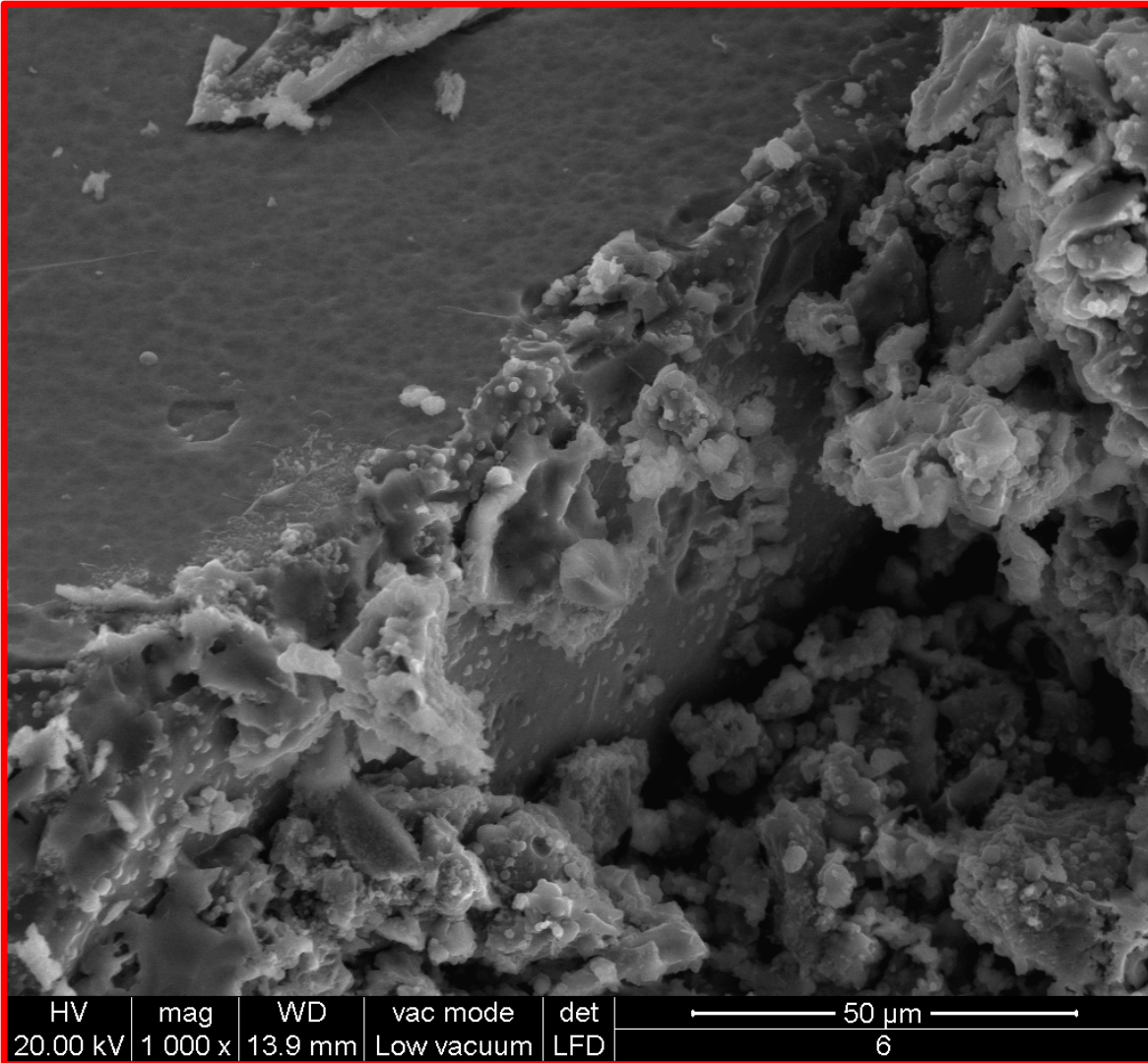


Binder from Acid Treated glass, polished surface

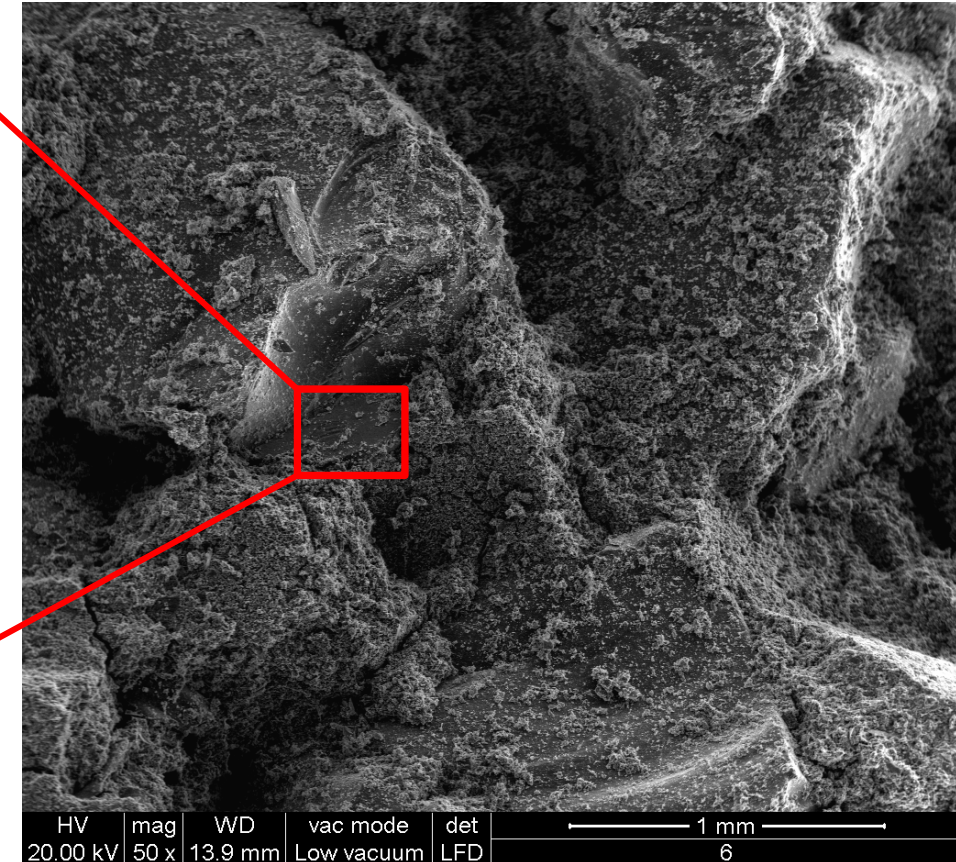


Extension: dilution with coarse glass

Glass-based concrete: enhancing sustainability
50wt% coarse glass added



Also 'free' glass surfaces showing some interaction with the binder



Extensions

Are there more 'geopolymer-oriented' waste glasses than SLG?

Can be activation 'lighter' and simpler?

Can we get products with extended functionalities?

Data in wt%

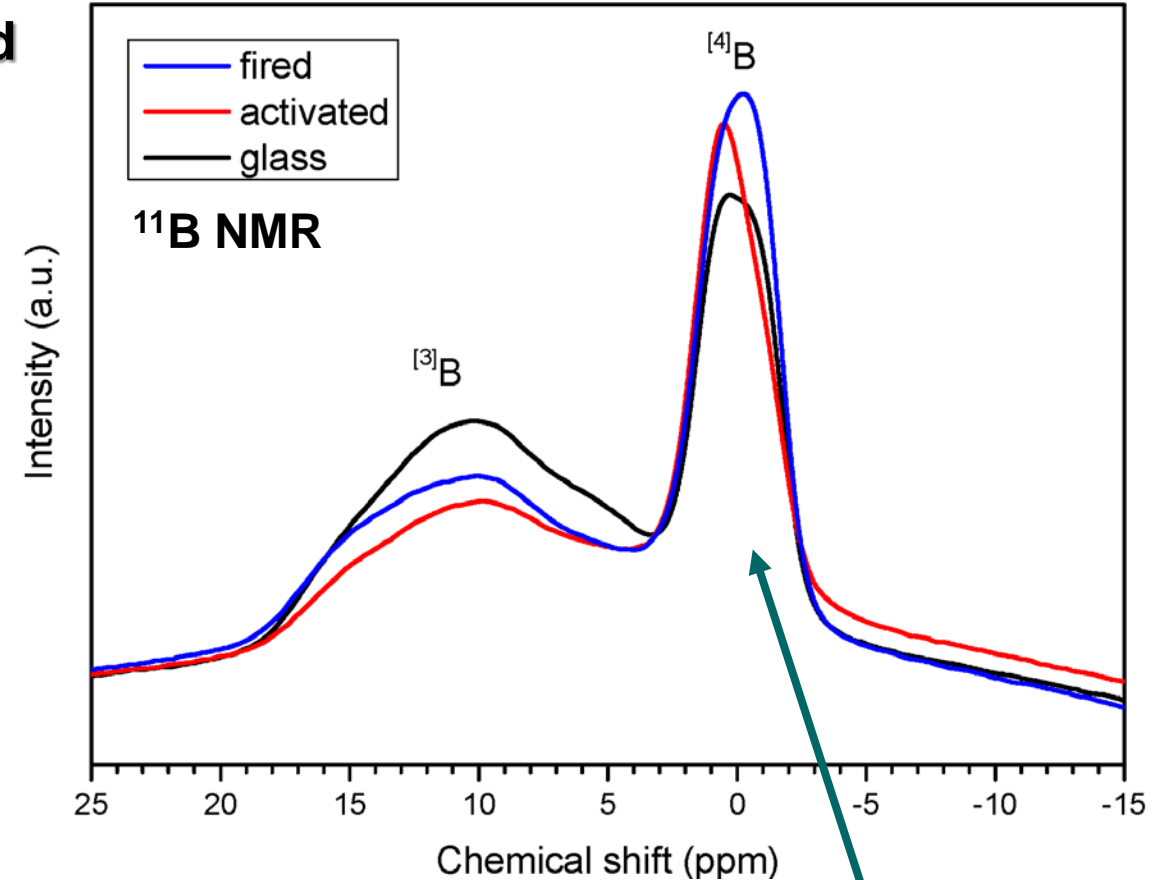
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SrO		
others	0.5	
L.O.I.		

Glass courtesy of Nuova Ompi – Stevanato Group (Padova, Italy)

N-A-S-H gel verified also in 'weak' aqueous solutions containing 2.5 M NaOH



See Rincon Romero et al. *Materials* 2018, 11(12), 2545

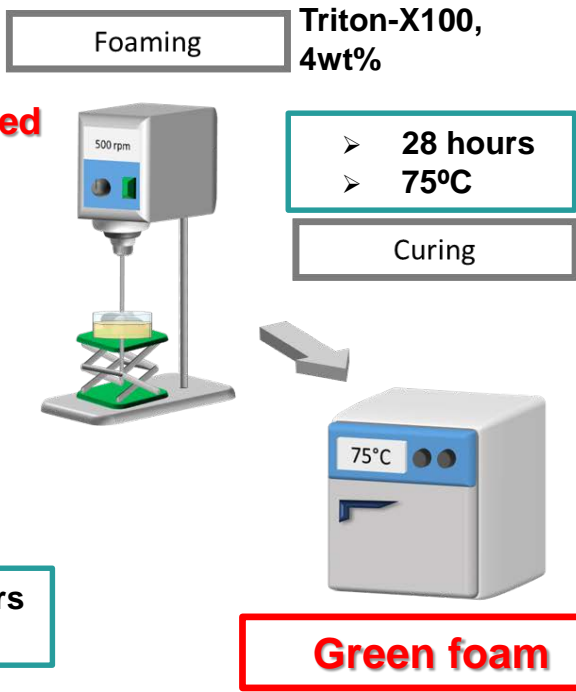


Evidence of enhanced BO_4 units, trapping Na^+ ions [in gel, confirmed even after firing at 800 °C]

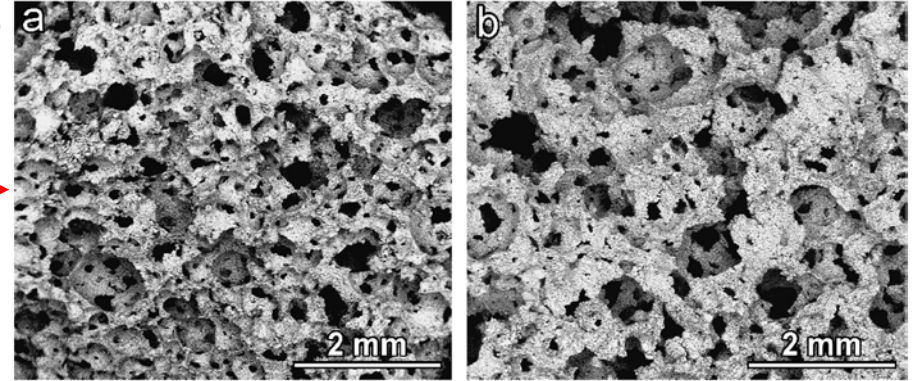
Article
Extension of the 'Inorganic Gel Casting' Process to the Manufacturing of Boro-Alumino-Silicate Glass Foams

Dissolution
 ➤ 4 hours
 NaOH or NaOH/KOH conc.
 ➤ 2.5M
 Solid loading
 ➤ 68 wt%

Mechanical foaming, surfactant-aided

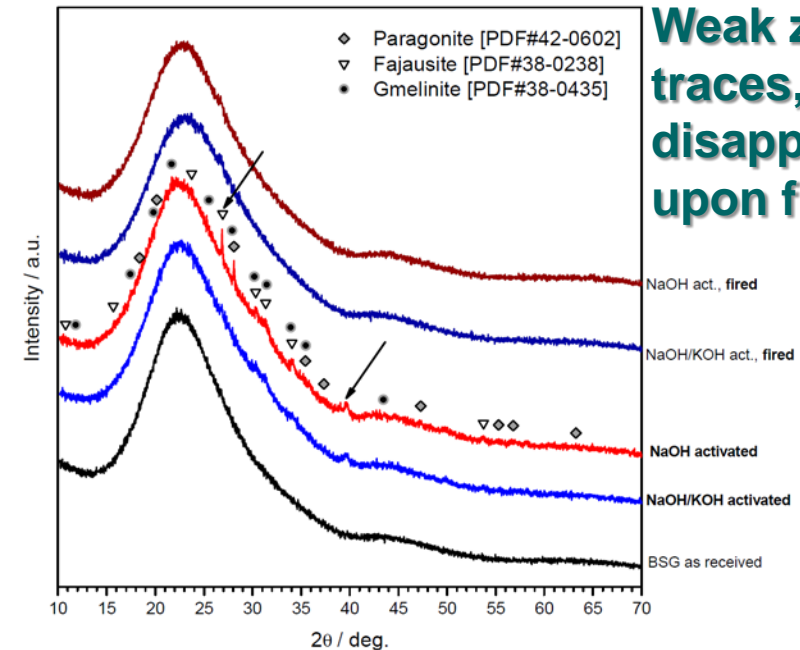


Particles bound by surface gel



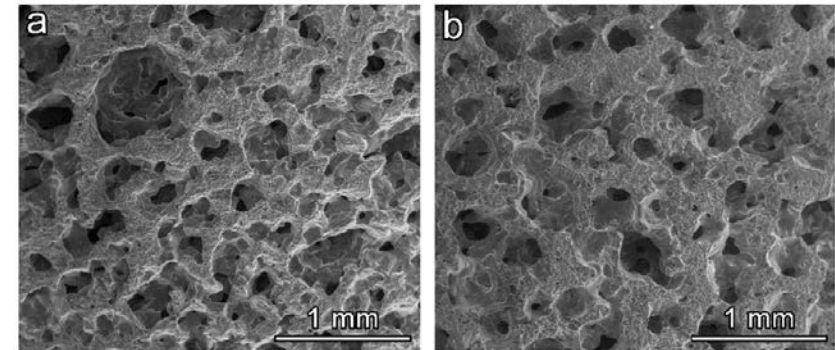
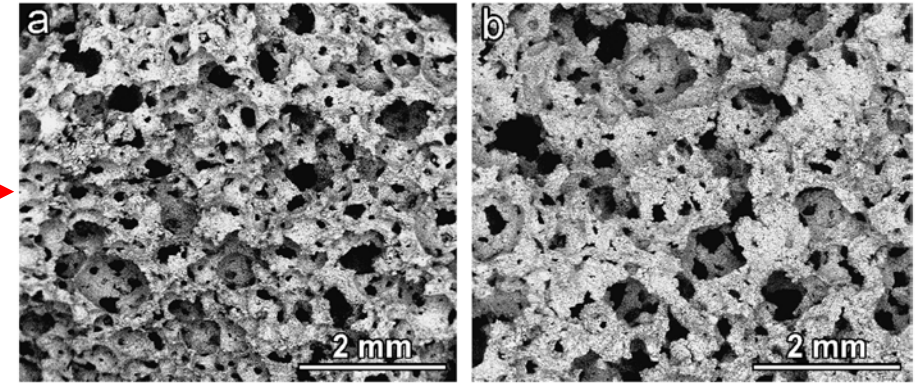
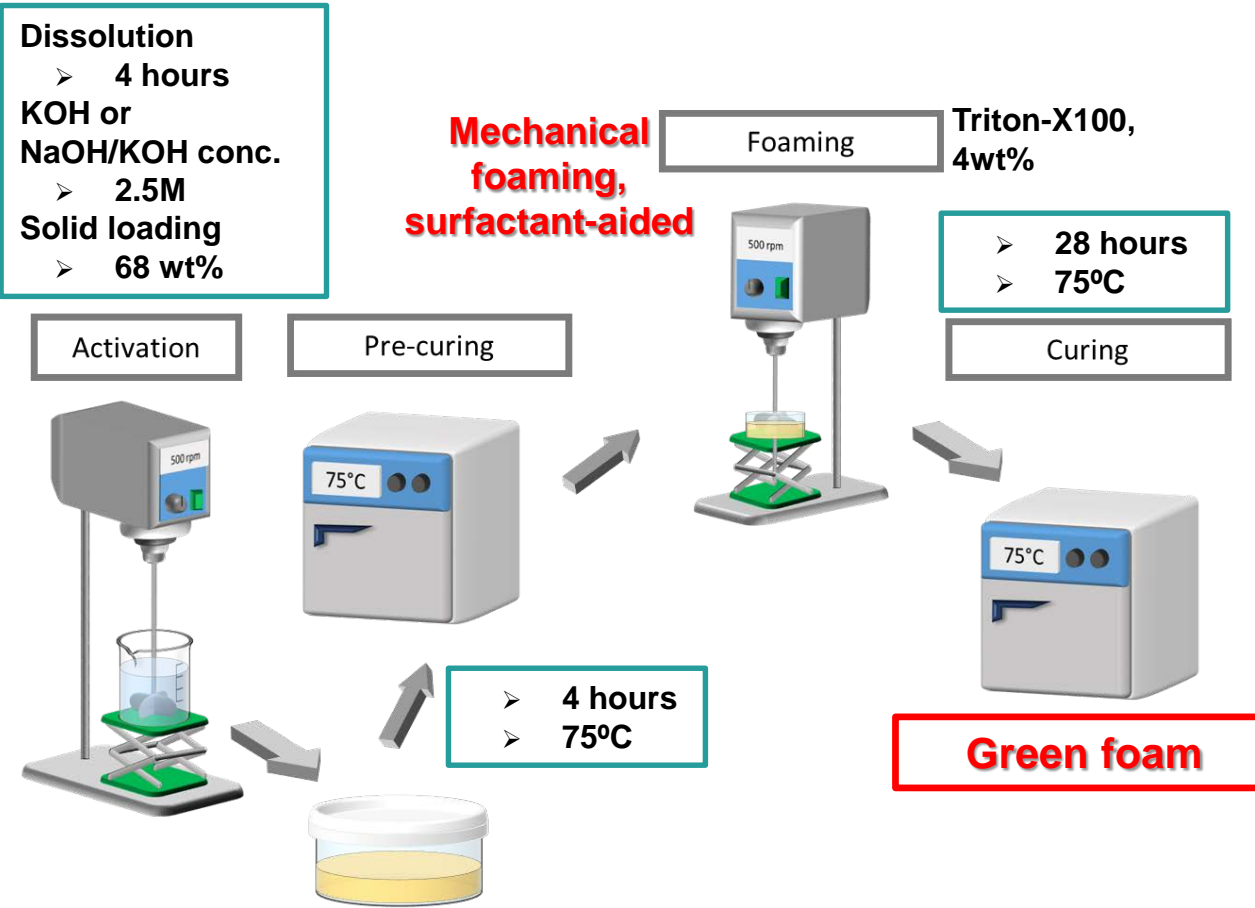
NaOH

NaOH/KOH (50mol%-50mol%)

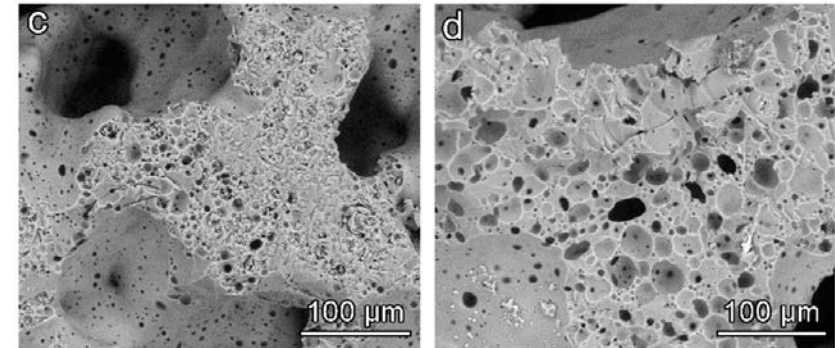


Weak zeolite traces, disappearing upon firing

Article
Extension of the 'Inorganic Gel Casting' Process to the Manufacturing of Boro-Alumino-Silicate Glass Foams



Fired at 700°C



Surfactant (%)	4			
Sintering Temperature (°C)	700			Green
ρ_{geom} (g/cm ³)	0.87 ± 0.03			0.58 ± 0.02
$\rho_{apparent}$ (g/cm ³)	1.88 ± 0.05			2.31 ± 0.02
ρ_{true} (g/cm ³)	0.38 ± 0.03			0.25 ± 0.02
Total Porosity (vol %)	61.6			75.1
Open Porosity (vol %)	53.7			74.8
Closed Porosity (%)	7.9			0.3
σ comp (MPa)	6.7 ± 0.4			0.5 ± 0.1

**Foams in the green state are weak
Consolidation after firing**

Surfactant (%)	4			
Sintering Temperature (°C)	700	650	550	Green
ρ_{geom} (g/cm ³)	0.87 ± 0.03	0.70 ± 0.03	0.54 ± 0.03	0.58 ± 0.02
$\rho_{apparent}$ (g/cm ³)	1.88 ± 0.05	2.17 ± 0.03	2.38 ± 0.04	2.31 ± 0.02
ρ_{true} (g/cm ³)	0.38 ± 0.03	0.30 ± 0.04	0.23 ± 0.03	0.25 ± 0.02
Total Porosity (vol %)	61.6	69.3	77.4	75.1
Open Porosity (vol %)	53.7	67.7	77.2	74.8
Closed Porosity (%)	7.9	1.7	0.2	0.3
σ_{comp} (MPa)	6.7 ± 0.4	3.9 ± 0.4	0.8 ± 0.2	0.5 ± 0.1

Foams in the green state are weak

Consolidation after firing at lower temperature: gel binder transforming into low-softening glass

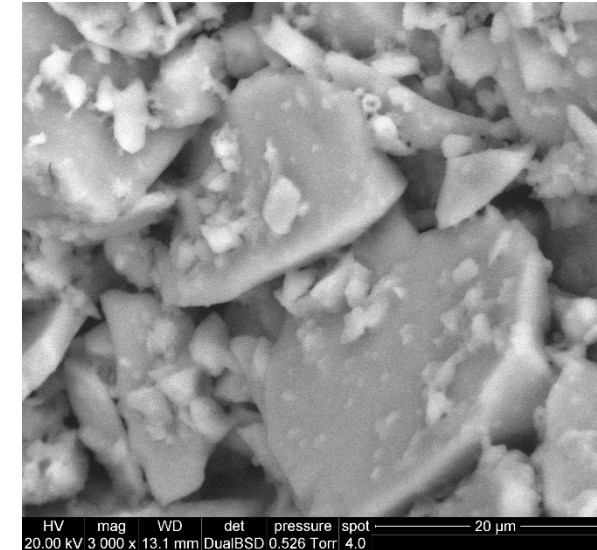
Surfactant (%)	4				2		
	Sintering Temperature (°C)	700	650	550	Green	650	550
ρ_{geom} (g/cm ³)	0.87 ± 0.03	0.70 ± 0.03	0.54 ± 0.03	0.58 ± 0.02	0.77 ± 0.03	0.64 ± 0.04	0.57 ± 0.03
$\rho_{apparent}$ (g/cm ³)	1.88 ± 0.05	2.17 ± 0.03	2.38 ± 0.04	2.31 ± 0.02	2.08 ± 0.04	2.36 ± 0.05	2.32 ± 0.04
ρ_{true} (g/cm ³)	0.38 ± 0.03	0.30 ± 0.04	0.23 ± 0.03	0.25 ± 0.02	0.34 ± 0.03	0.27 ± 0.04	0.24 ± 0.02
Total Porosity (vol %)	61.6	69.3	77.4	75.1	66.4	78.9	76.0
Open Porosity (vol %)	53.7	67.7	77.2	74.8	62.8	78.8	75.4
Closed Porosity (%)	7.9	1.7	0.2	0.3	3.6	0.1	0.6
σ_{comp} (MPa)	6.7 ± 0.4	3.9 ± 0.4	0.8 ± 0.2	0.5 ± 0.1	2.1 ± 0.2	0.7 ± 0.1	0.7 ± 0.1

Confirmation with lower surfactant content

From glass foams to membranes

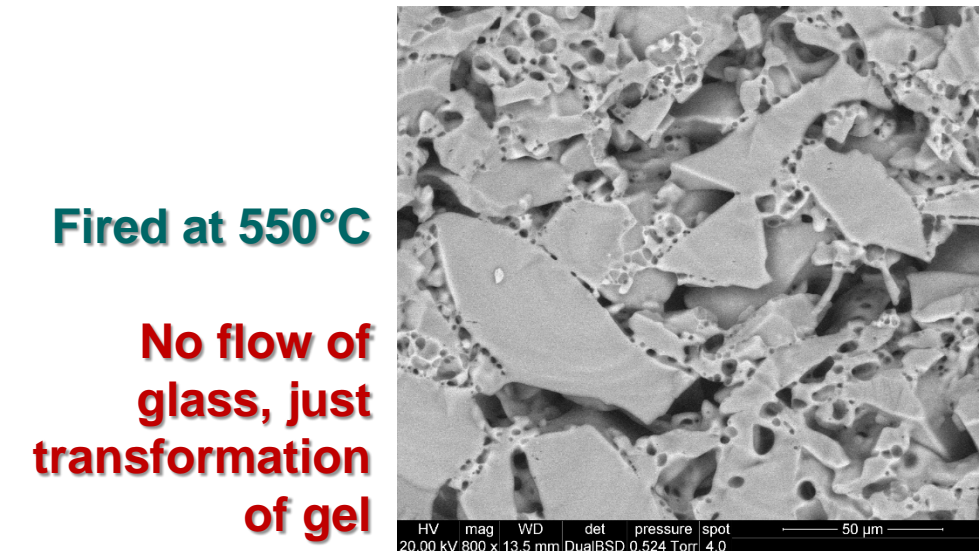
Enhanced strength with no foaming
Attention to binding phase

Surfactant (%)	4				No surfactant		
Sintering Temperature (°C)	700	650	550	Green	650	550	Green
ρ_{geom} (g/cm ³)	0.87 ± 0.03	0.70 ± 0.03	0.54 ± 0.03	0.58 ± 0.02	1.49 ± 0.03	1.41 ± 0.03	1.45 ± 0.04
$\rho_{apparent}$ (g/cm ³)	1.88 ± 0.05	2.17 ± 0.03	2.38 ± 0.04	2.31 ± 0.02	2.01 ± 0.03	2.19± 0.03	2.38 ± 0.03
ρ_{true} (g/cm ³)	0.38 ± 0.03	0.30 ± 0.04	0.23 ± 0.03	0.25 ± 0.02	0.59 ± 0.05	0.65 ± 0.05	0.61 ± 0.04
Total Porosity (vol %)	61.6	69.3	77.4	75.1	41.2	34.7	39.0
Open Porosity (vol %)	53.7	67.7	77.2	74.8	31.3	31.9	39.0
Closed Porosity (%)	7.9	1.7	0.2	0.3	9.9	2.8	0.0
σ_{comp} (MPa)	6.7 ± 0.4	3.9 ± 0.4	0.8 ± 0.2	0.5 ± 0.1	19.4 ± 0.4	16.4 ± 0.5	24.4 ± 0.6



**Green,
unfoamed
Gel binding
particles**

**No foaming:
more contact**



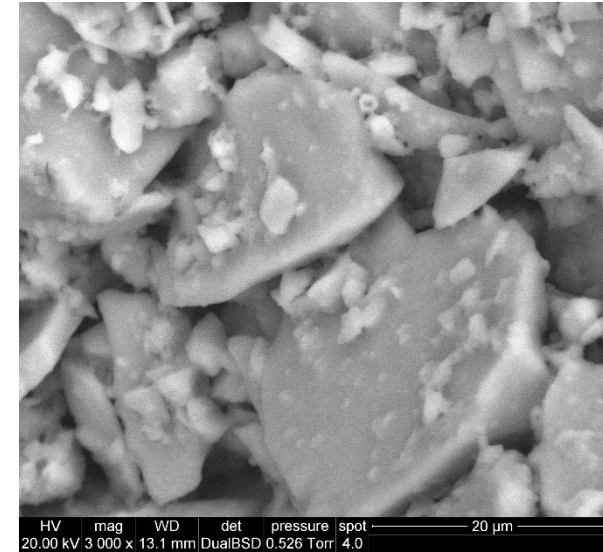
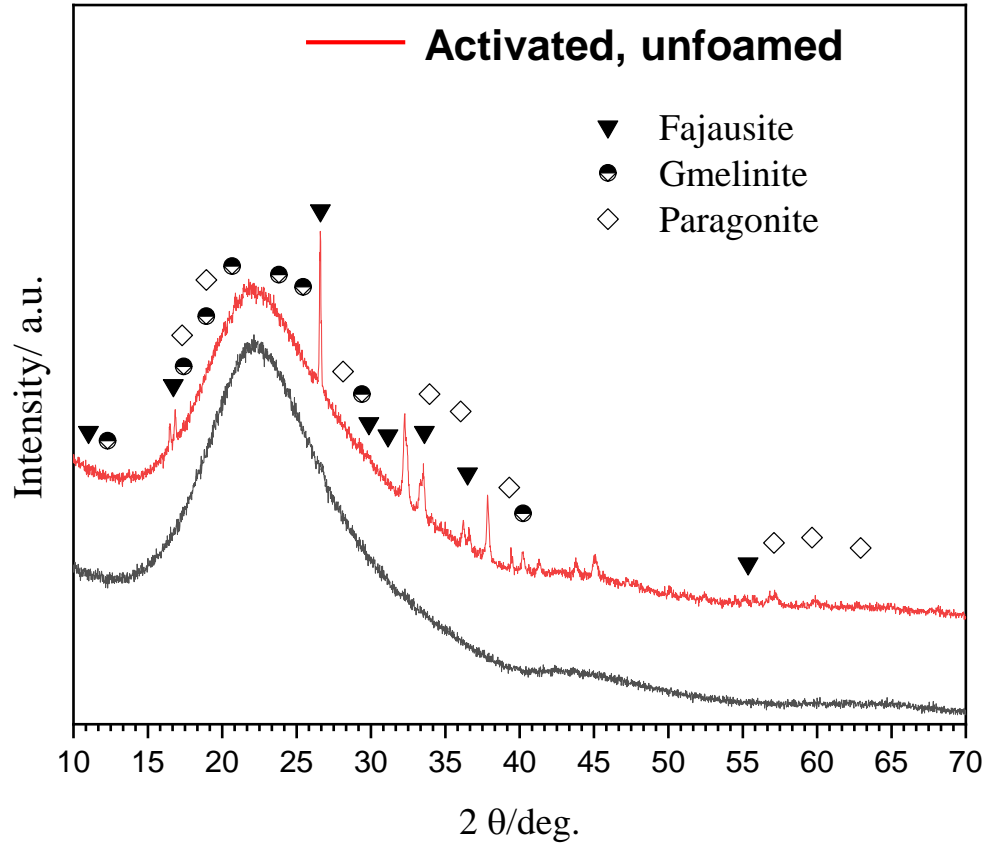
Fired at 550°C

**No flow of
glass, just
transformation
of gel**

From glass foams to membranes

Enhanced strength with no foaming
Attention to binding phase

Enhanced
zeolite
formation
→ Use in water
purification

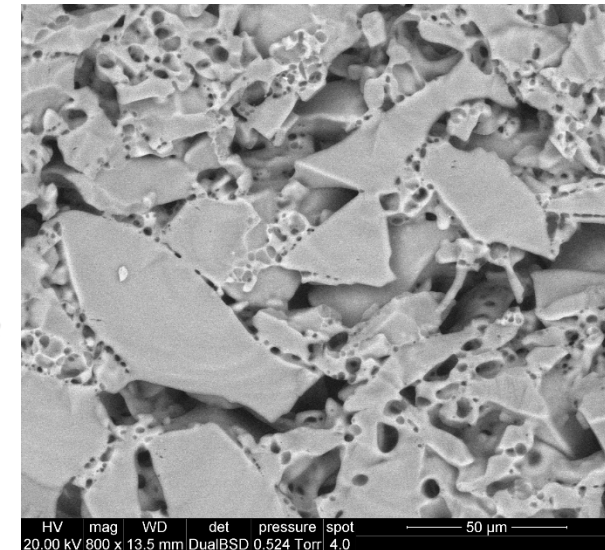


**Green,
unfoamed
Gel binding
particles**

**No foaming:
more contact**

Fired at 550°C

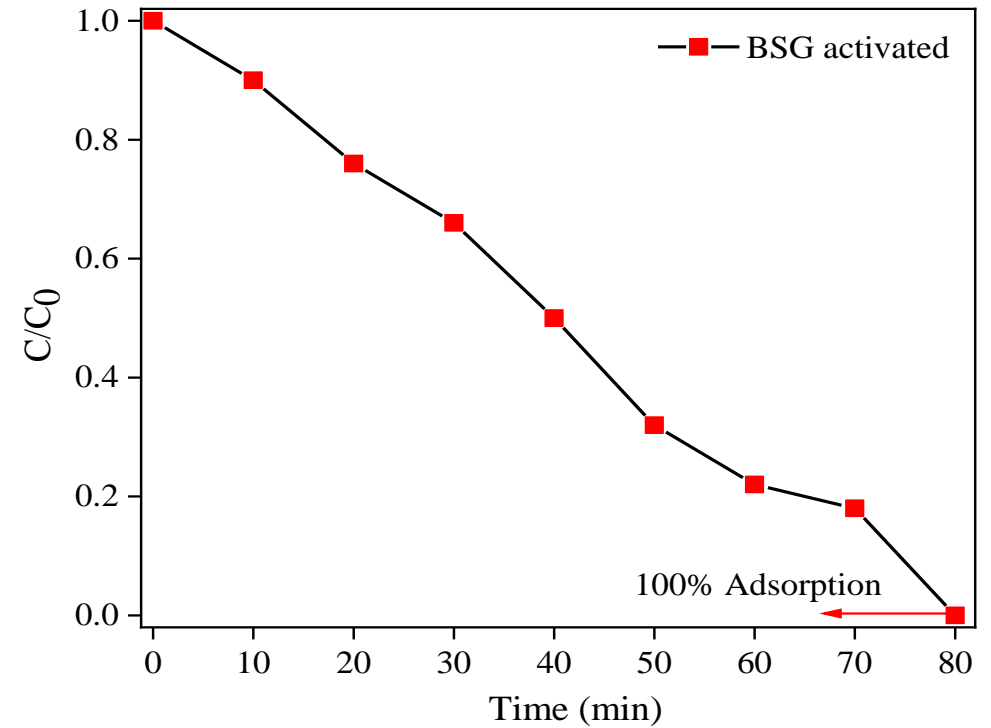
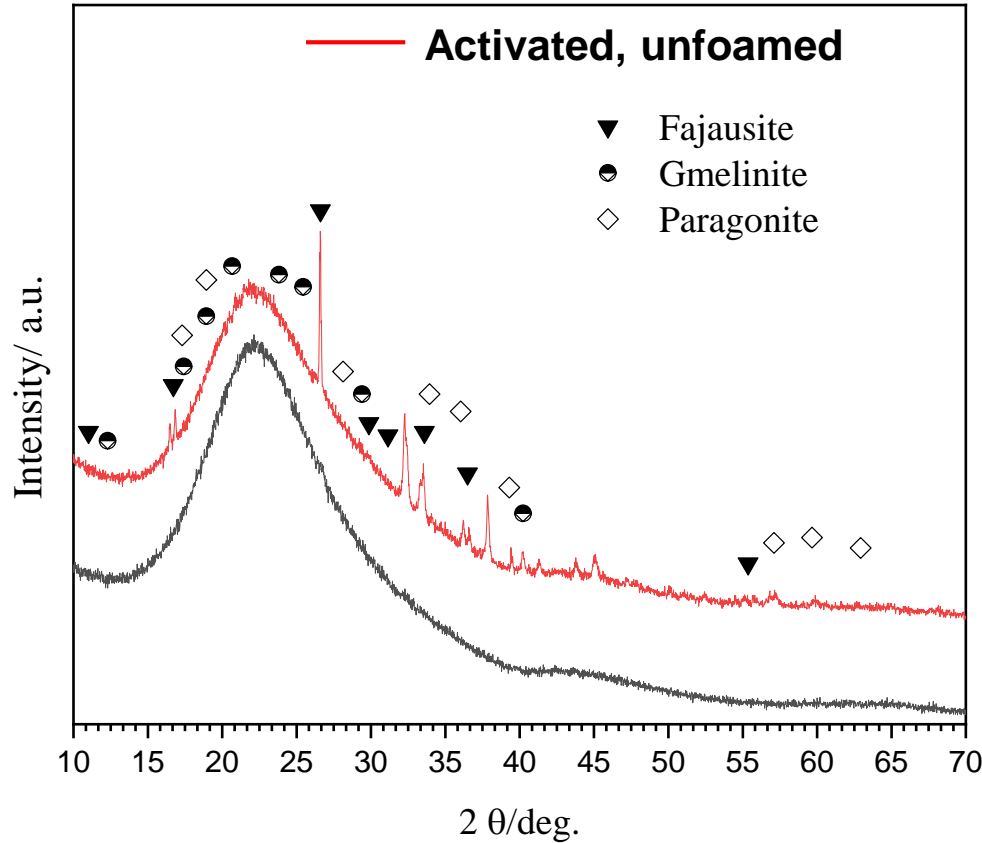
**No flow of
glass, just
transformation
of gel**



From glass foams to membranes

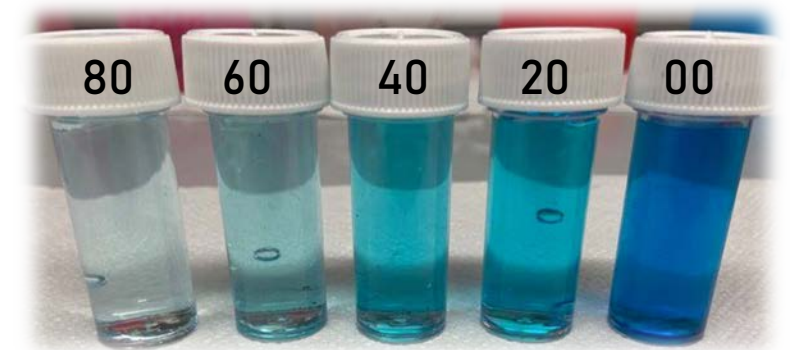
Enhanced strength with no foaming
Attention to binding phase

Enhanced
zeolite
formation
→ Use in water
purification



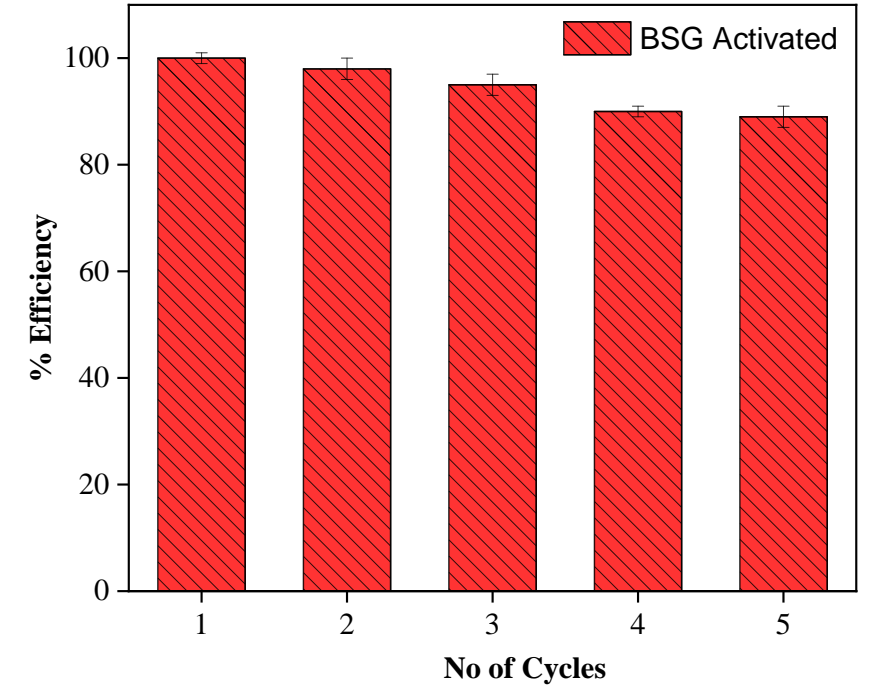
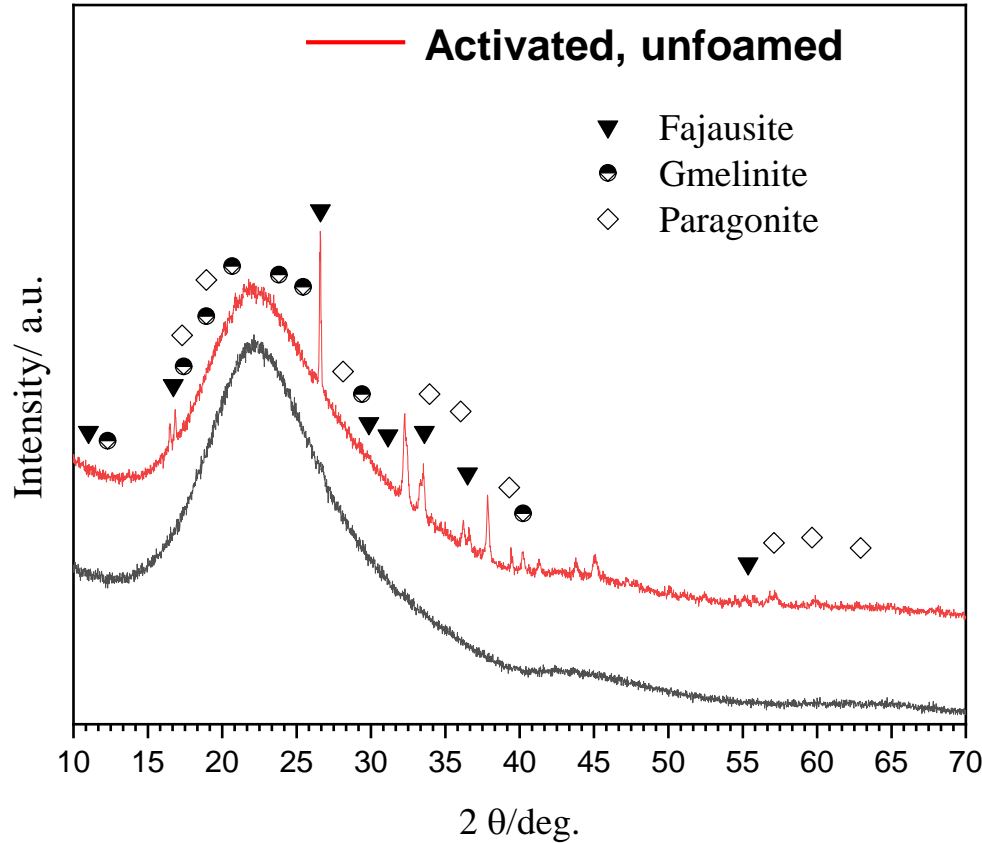
Application in Dye Removal from Water

Model Dye: Methylene Blue, Initial concentration: 10mg/mL



In progress

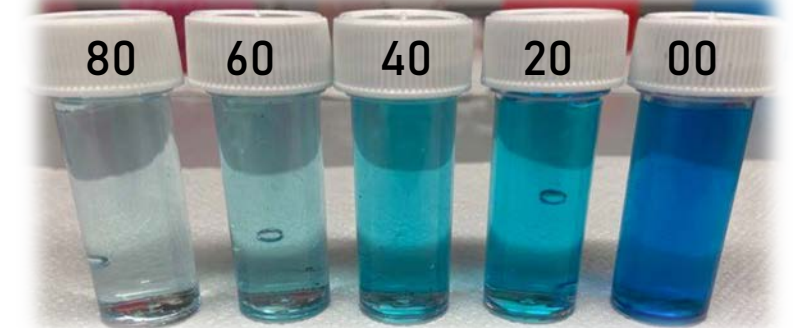
Enhanced zeolite formation
→ **Use in water purification**



Application in Dye Removal from Water

Model Dye: Methylene Blue, Initial concentration: 10mg/mL

Reusable sorbents [1 cycle: 80 min adsorption + dye extraction by centrifugation]



BSG 'naturally' prone to zeolitization
But SLG can be combined with Al₂O₃ rich waste...

Chemical Component	Rock Wool (RW)
CaO	17.4
SiO ₂	40.4
Al ₂ O ₃	15.8
Fe ₂ O ₃	9.2
Na ₂ O	1.4
K ₂ O	0.4
MgO	12.6
P ₂ O ₅	0.1
TiO ₂	0.8
SO ₃	n.d
Cl	n.d

Raw materials: 50 wt% SLG, 50 wt% RW (all amorphous)

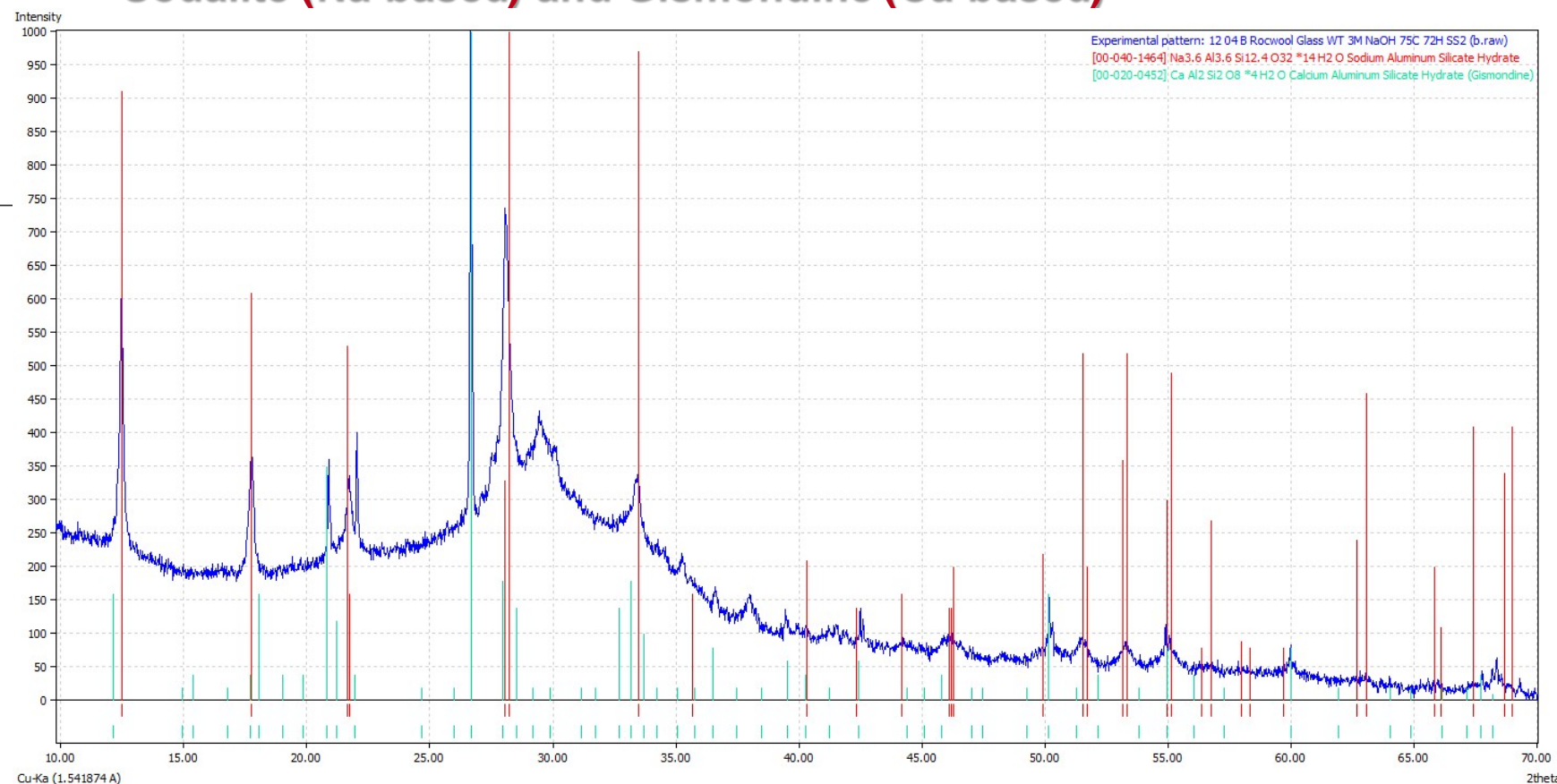
In solution 3M NaOH (30 min), 3d at 75 °C

Liq/Sol=0.53

Activation: formation of zeolites

Sodalite (Na-based) and Gismondine (Ca-based)

J. Yliniemi et al., Utilization of Mineral Wools as Alkali-Activated Material Precursor. *Materials* 9 (2016) 312.



Back to SLG: cold consolidation and new foams

While BSG is 'naturally' prone to zeolitization
SLG can still be combined with Al₂O₃ rich waste...

Chemical Component	Rock Wool (RW)
CaO	17.4
SiO ₂	40.4
Al ₂ O ₃	15.8
Fe ₂ O ₃	9.2
Na ₂ O	1.4
K ₂ O	0.4
MgO	12.6
P ₂ O ₅	0.1
TiO ₂	0.8
SO ₃	n.d
Cl	n.d

J. Yliniemi et al., Utilization of Mineral Wools as Alkali-Activated Material Precursor. *Materials* 9 (2016) 312.

After drying
Sample surviving boiling test
[geopolymer-like]

Raw materials: 50 wt% SLG, 50 wt% RW (all amorphous)

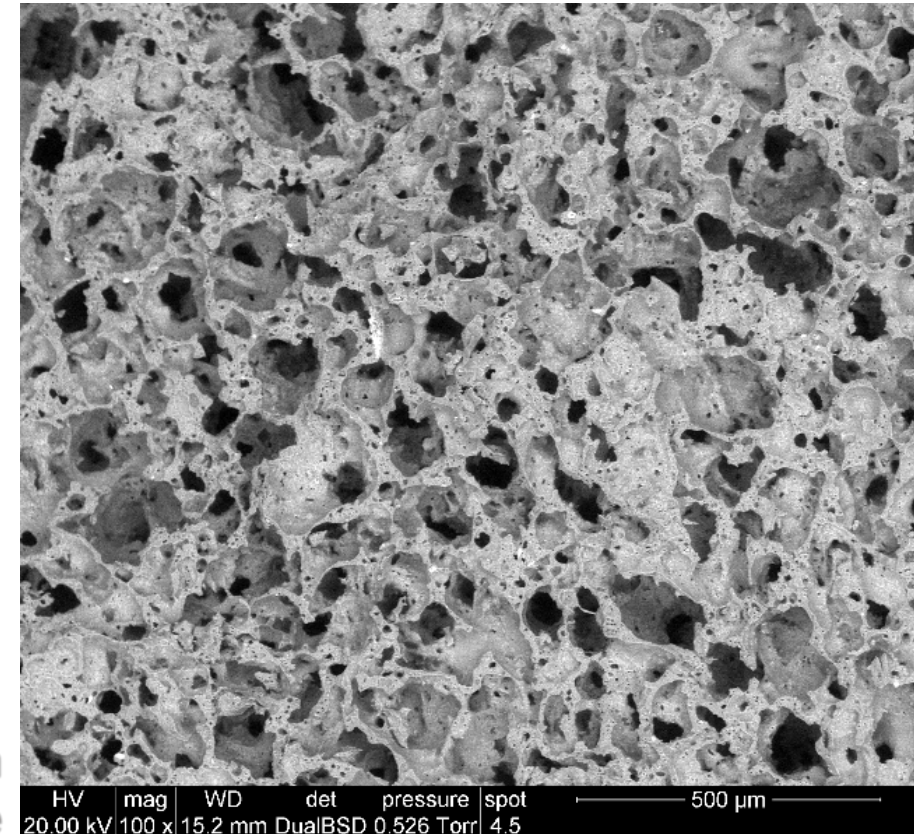
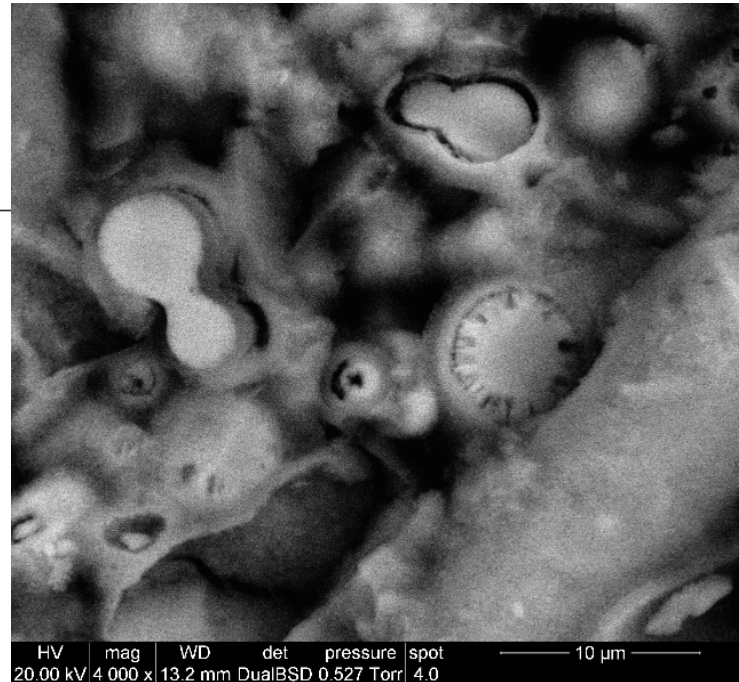
In solution 3M NaOH (30 min), 3d at 75 °C

Liq/Sol=0.53

Activation: formation of zeolites

Sodalite (Na-based) and Gismondine (Ca-based)

In progress



After firing at 800 °C: glass-ceramic foam
Foaming by decomposition of hydrated binding phase

Discarded glasses expressing great potential for the obtainment of stable inorganic gels

Systems to be adjusted by:

- **Activators:** Aluminates significant in yielding AlO_4 units
- **Selection of glasses:** some glasses are inherently prone to the formation of zeolite-like gels
- **Secondary waste:** attention to Al_2O_3 -rich waste

Other features:

- **Development of porous bodies:** exploration of pore generation at the early stage of gelation (by intensive **mechanical stirring**) or **decomposition of binding phase**
- **Sorbents:** **simplified approach, focus on zeolite phases**

- **European Community's Horizon 2020 Programme** through a Marie Skłodowska-Curie Innovative Training Network (**'CoACH'**, g.a. no. 642557) <http://www.coach-etn.eu>
- European Community's Horizon 2020 Programme through H2020-WIDESPREAD-01-2016-2017-TeamingPhase2 project (**'FunGLASS'**, g.a. no. 739566) <http://www.funglass.eu/>



MANY THANKS FOR YOUR ATTENTION!

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<http://www.dii.unipd.it/bernardo>