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I INTERNATIONAL SYMPOSIUM "CALCINED CLAYS AS SUPPLEMENTARY CEMENTITIOUS MATERIALS"

Assessment of the pozzolanic reactivity of calcined kaolinitic clays by a rapid alkaline solubility test

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SIQ 2016 Sociedad Cubana de Química

Cuba, June 2016

CIDem centro de investigación y desarrollo de estructuras y materiales

ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE

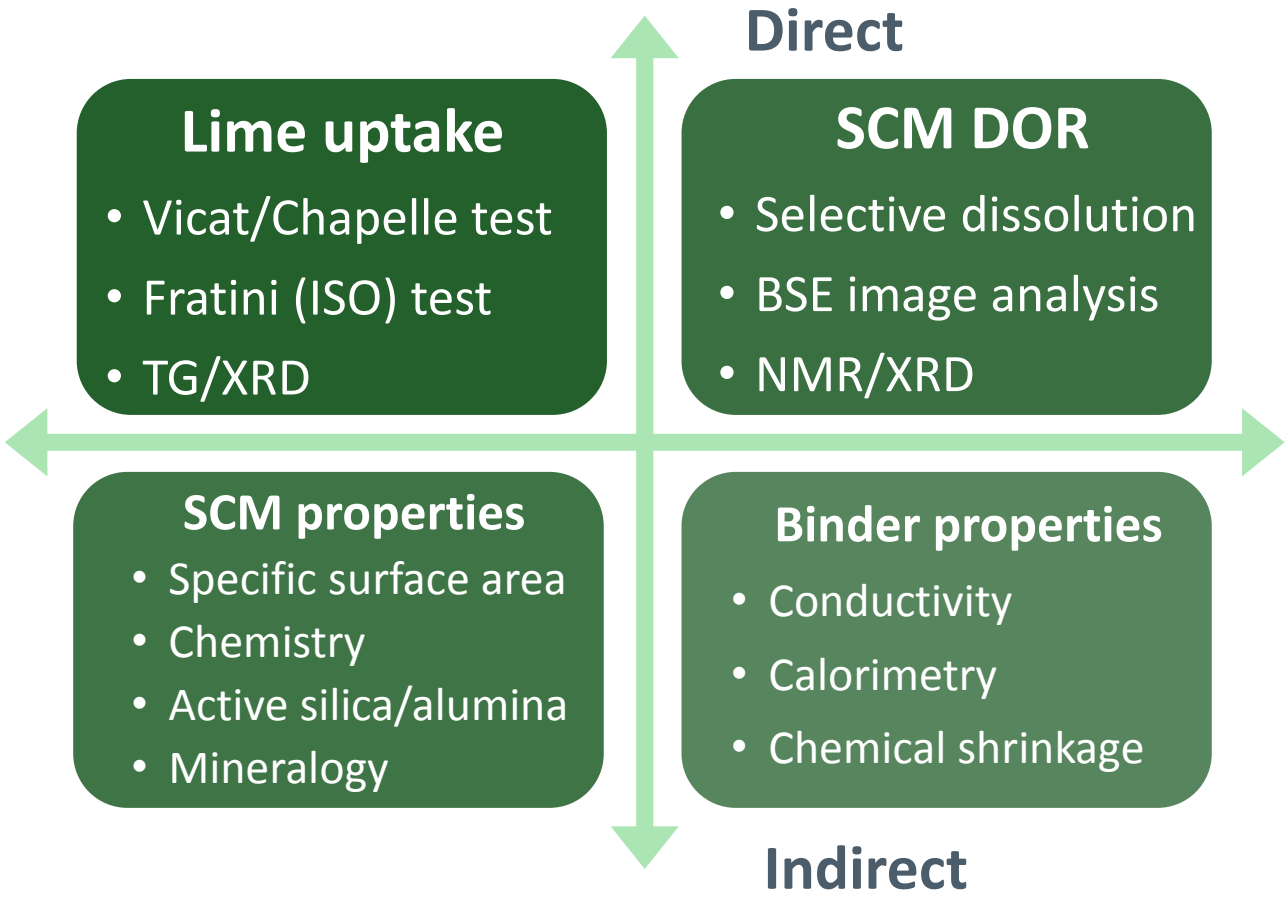
Introduction: Clays as a source of SCM

- Growing interest in the use of calcined clays as Supplementary Cementitious Materials (SCM).

- Natural clays exhibits a great chemical and mineralogical complexity which influence the performance of its calcination products as SCM.

Evaluate the pozzolanic reactivity of calcined kaolinitic clays by rapid and reliable tests

Existing methods classified according to measured property

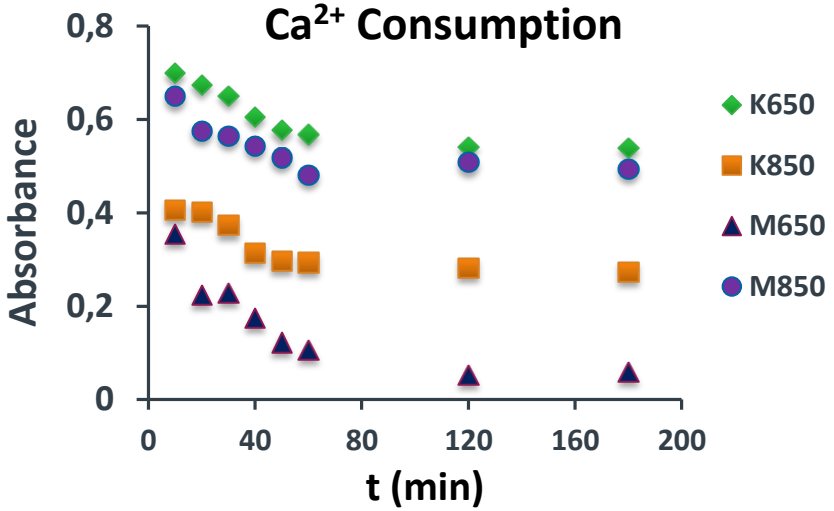


Slide from R. Snellings, 2014.

Drawbacks of existing methods

Lime uptake

Tests based on portlandite consumption may be interfered by other phenomena no related to pozzolanic reactivity (cation exchange?)



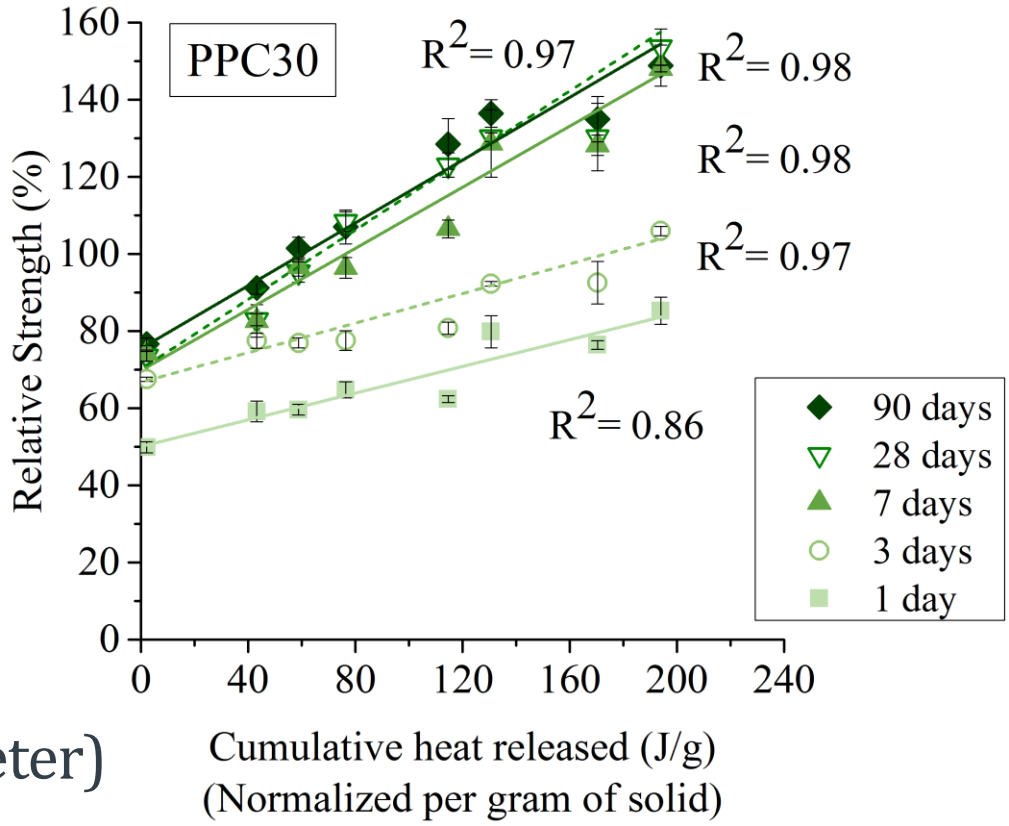
Mechanical strength

- Long testing times are required
- Filler effect must be taking into account

R3 Test (Isothermal Calorimetry)

Raw material → **Grinding** → **Calcination at 850 °C**

Rapid, reliable and relevant test. Good correlation with compressive strength.

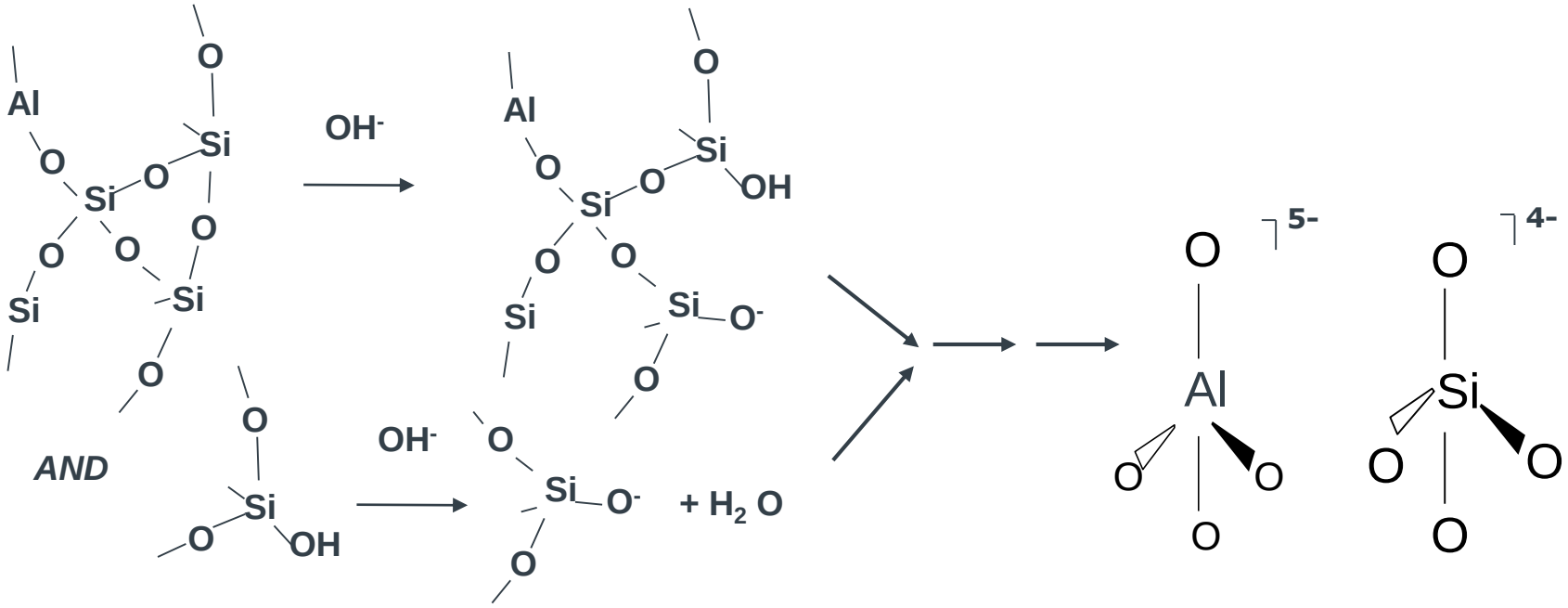


(Requires an isothermal calorimeter)

(Avet et al., 2015)

Pozzolanic Reactivity & Alkaline Solubility

Pozzolanic reactivity must be directly proportional to the amount of soluble silicon and aluminum species released in an alkaline solution with a pH similar to the hydrated cement pore solution.



LS Dent Glasser and N Kataoka, Cem Concr Res, 12, 321, (1982)

Dissolution treatment of pozzolans: active silica and alumina

Method	Dissolution procedure	Reference
HNO_3/KOH	1. HNO_3 concentrated 2. 10% KOH (12h)	Rivot, 1862
HCl/KOH	1. 20% HCl (cold) 2. 20% KOH (cold, 20h) 3. 20% KOH (50-65 °C, 4h)	Baire, 1930
HCl/NaOH	1. 50% HCl (hot) 2. NaOH (hot)	Malquori, 1935
NaOH/HCl	1. NaOH (1 N, hot, 0.5h) 2. 50% HCl (hot) (2x)	ASTM C379-56T
HCl/Na_2CO_3	1. HCl (conc., heated) 2. Na_2CO_3 +NaCl (hot, 15 min)	AFNOR P 15-301
Na_2CO_3 + NaOH	3.6% Na_2CO_3 + 1% NaOH (5 min, repeated X times)	Steopoe, 1956
Salicylic acid + methanol	Salicylic acid (25 g) in 300 ml methanol	Takashima, 1958
HF + HNO_3	2 M HF + 0.6 M HNO_3 (1h) Heat evolution measured by calorimetry	Jambor, 1962
HF	1 M HF (10 min, 300 K) Solution conductivity measured	Rhaask and Bhaskar, 1975

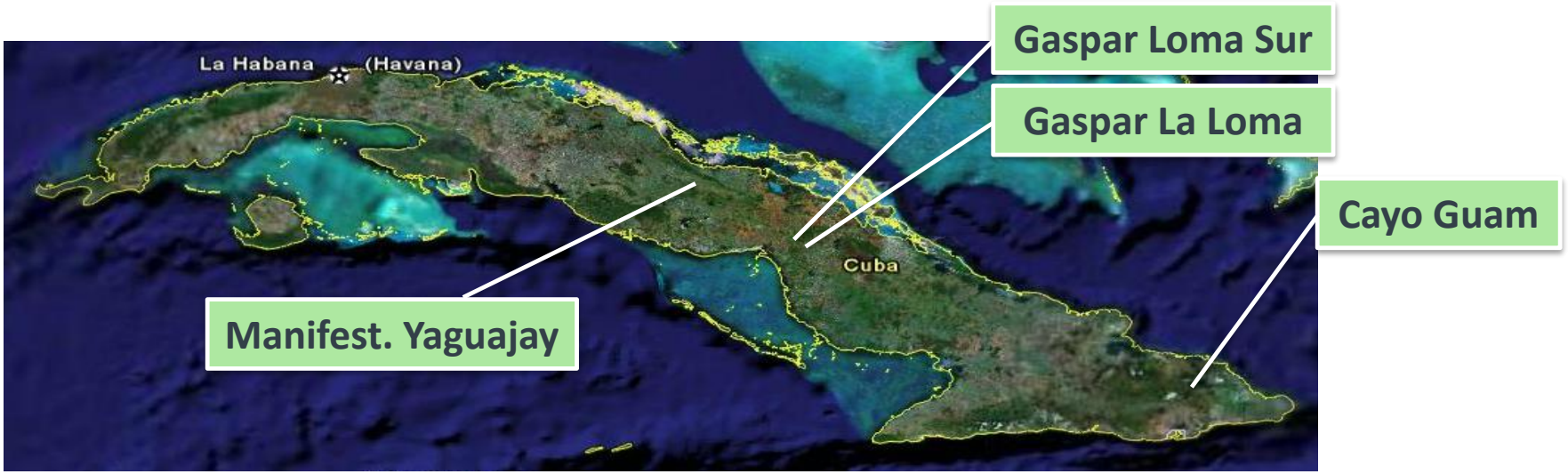
Existing methods are based on too harsh conditions

Slide from R. Snellings, 2014.

Dissolution treatment of pozzolans: active silica and alumina

Development of a rapid method to assess the pozzolanic reactivity of calcined clays based on its alkaline solubility

Selection of the raw material



Variability:

- Chemical
- Mineralogical
- Formation of the deposit

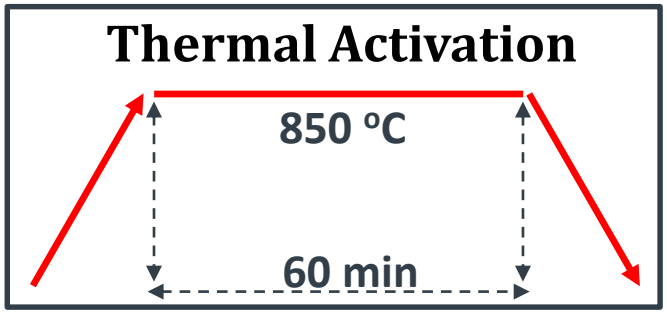
Characterization of the raw materials (XRF, XRD, TGA)

Clay	% SiO_2	% Al_2O_3	% Fe_2O_3	% CaO	Al_2O_3/SiO_2	% Keq
MK	51,61	40,44	1,52	0,32	0,78	89,20
CG	39,55	31,58	12,68	0,05	0,80	81,15
LS	50,88	25,23	16,97	0,28	0,50	57,85
LL	61,4	18,86	12,58	0,07	0,30	42,22
YG	46,58	20,06	9,61	**2,94**	0,43	60,14

Clay	Origen	1:1 Clay Minerals	2:1 Clay Minerals	Others
CG	Weathering	Halloysite, Kaolinite		Quartz and iron oxides and hydroxides (Fe_2O_3 and α-FeO(OH))
LS	Hydrothermal/ Weathering	Halloysite, Kaolinite	Vermiculite, Montmorillonite	
LL	Hydrothermal/ Weathering	Kaolinite, Nacrite		
YG	Redeposited	Nacrite, Kaolinite	Vermiculite	**+ Calcite (5,25%)**

- Equivalent kaolinite content is between 40 % (LL) and 80 % (CG)
- Predominance of clays type 1:1
- Presence of relative high content of calcite in YG deposit

Sample processing



Grinding

Calcination

Crushing and homogenization

90% passed through the sieve of 90 µm

Study of pozzolanic activity

Alkaline solubility

Isothermal Calorimetry (R3 test)

(Avet et al., 2015)

Methodology

50 mL NaOH (0,5M) + 0,1g calcined clay

50 ºC y 30 rpm / 24 hours

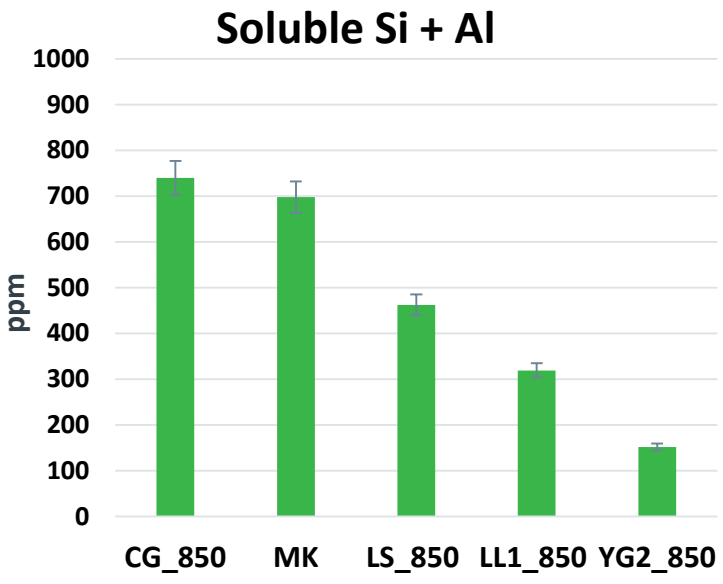
→ 25mL of dissolution + 2,5mL HCl (37%)

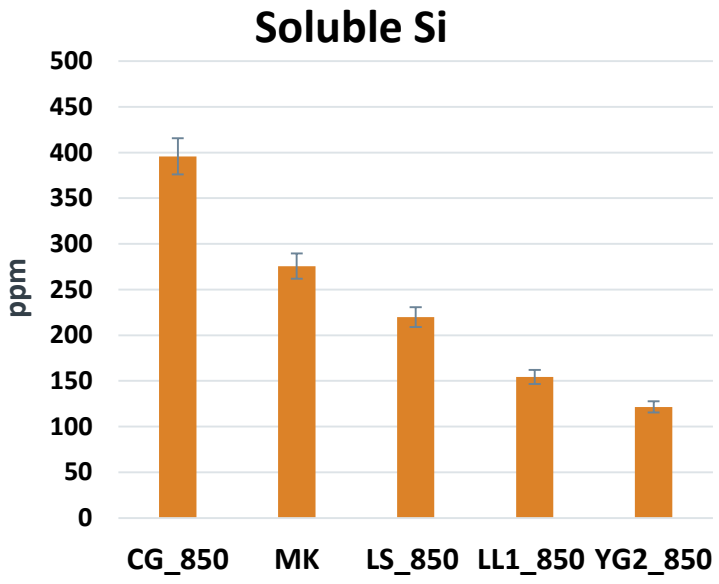
↓

- Centrifuge at 1500 rpm / 20 minutes
- Filtering with filtration paper

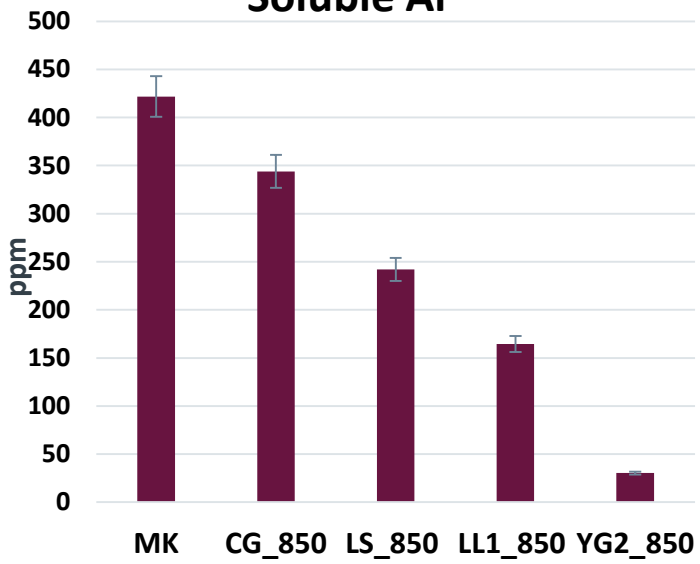
→ Atomic Absorption Spectroscopy

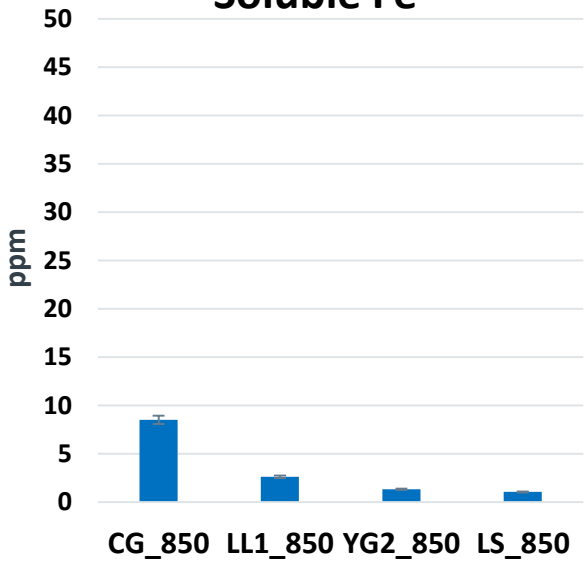
Results: Alkaline Solubility





Negligible solubility of Fe





Alkaline solubility

Soluble Si+Al

CG > MK > LS > LL > YG

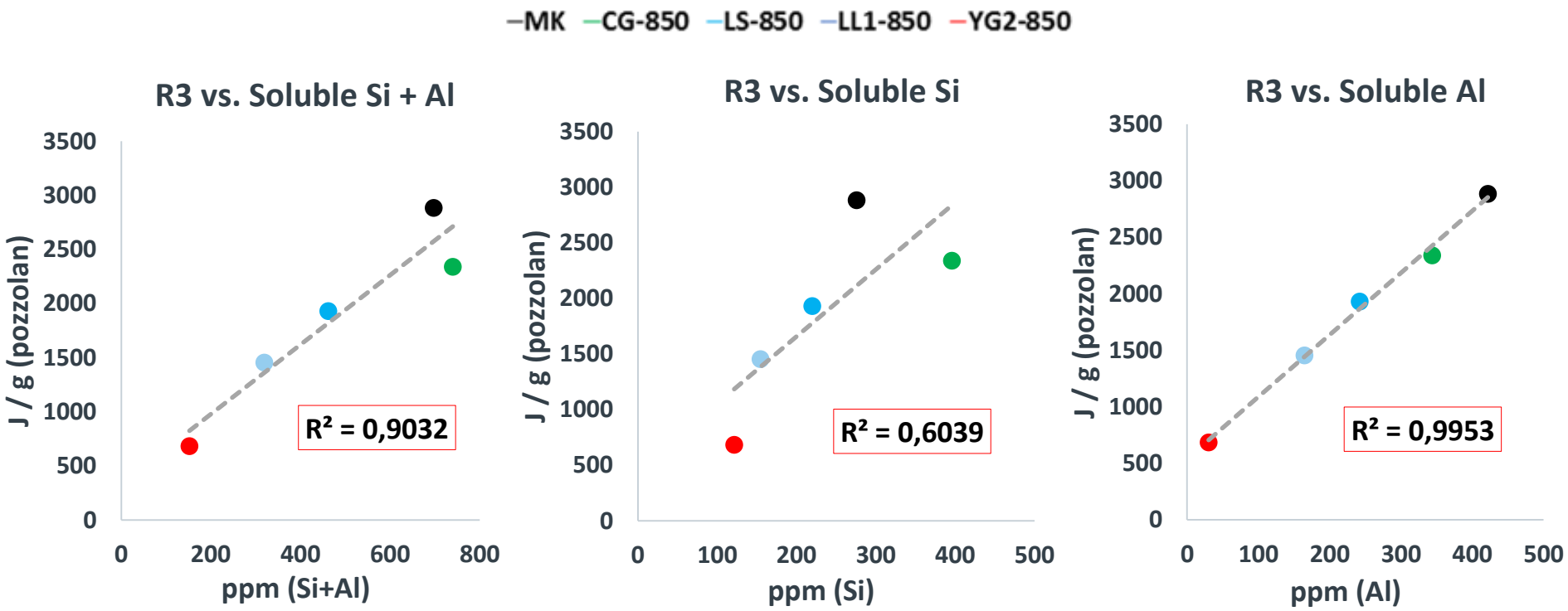
Soluble Si

CG > MK > LS > LL > YG

Soluble Al

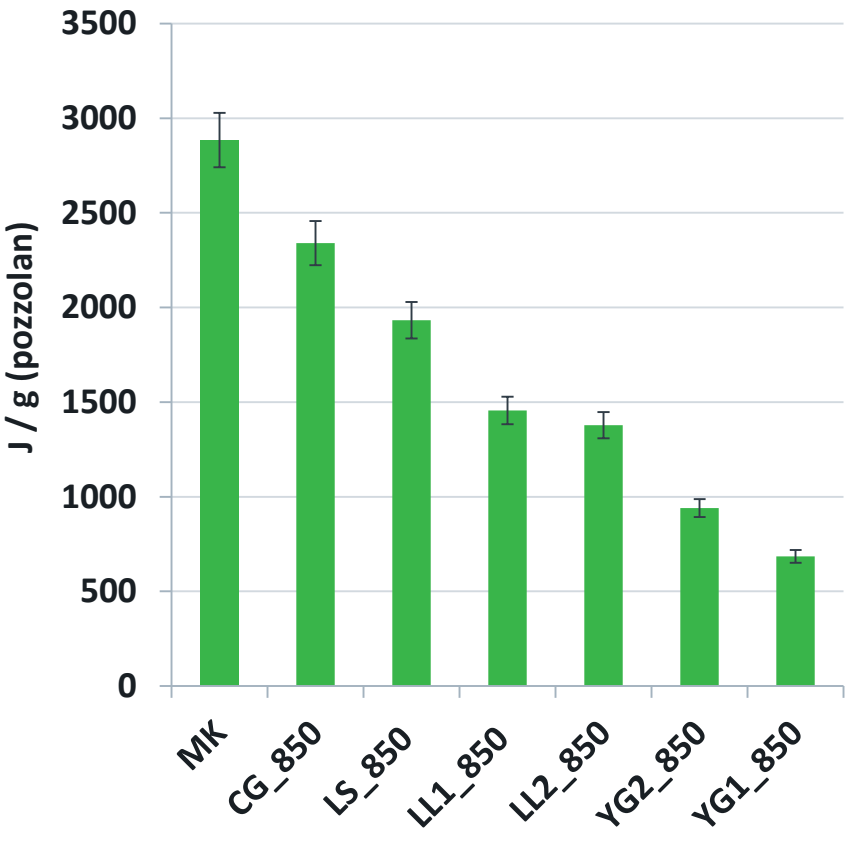
MK > CG > LS > LL > YG

Possible Correlation

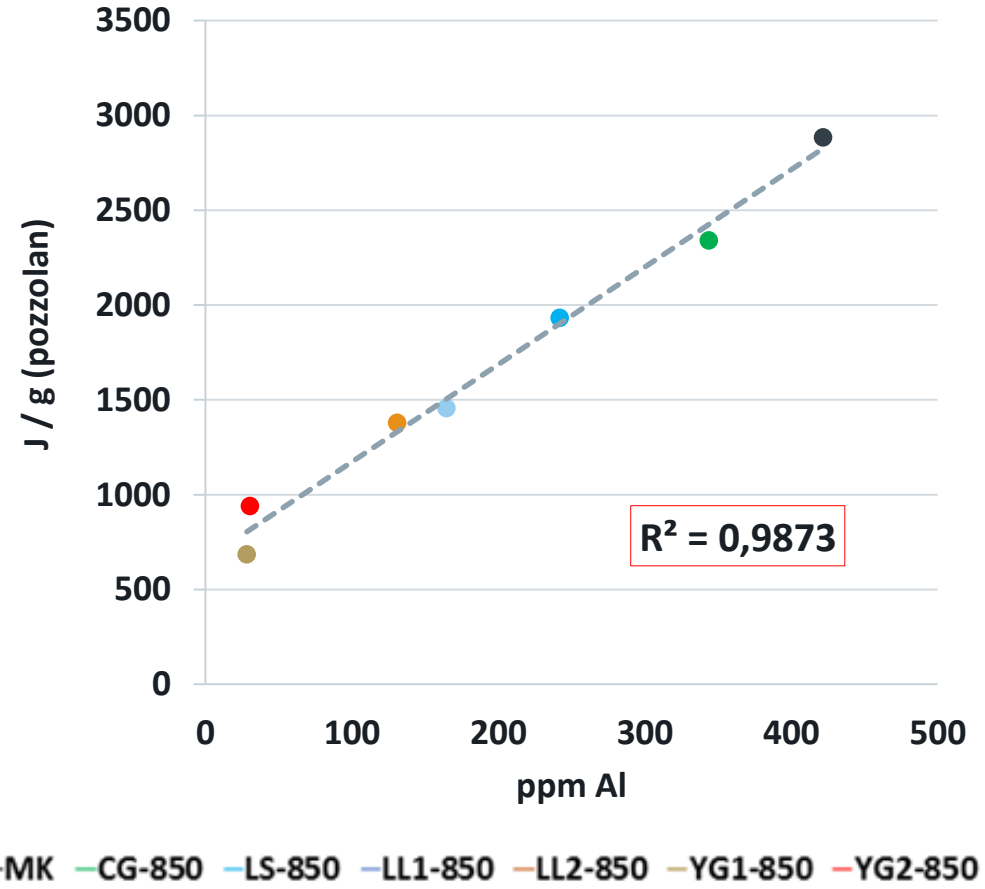


- For kaolinitic clays, correlation between R3 test and soluble Si and Si + Al is not good (low correlation coefficient)
- For kaolinitic clays, correlation with soluble Al is higher (the total accumulated heat comes largely from the reactions of the aluminates?)

Cummulative Heat (120 h)



R3 vs. Soluble Al



Final remarks

- For kaolinitic clays, there is a direct and good correlation between soluble aluminum and the total heat released by R3 test.

- Alkaline solubility test could be a viable test to evaluate the pozzolanic reactivity of calcined kaolinitic clays.

- Increase the number of calcined clays but with different conditions of calcination.

- Assessment of the pozzolanic reactivity of others pozzolans but they should be different of calcined clays (zeolite, silica fume, fly ash).

Thank you very much!

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