Reactivity Tests for Emerging SCMs: Towards Standardization and Specification

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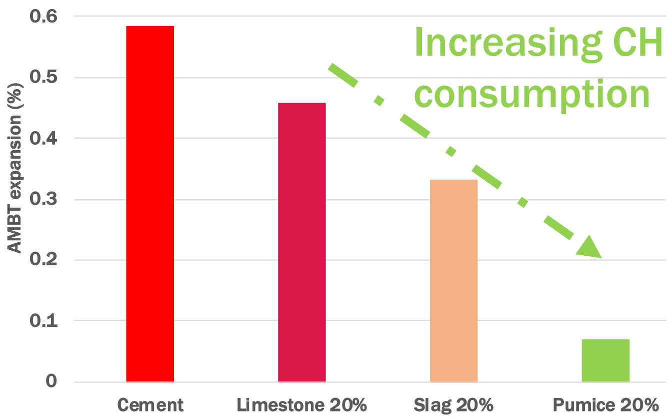
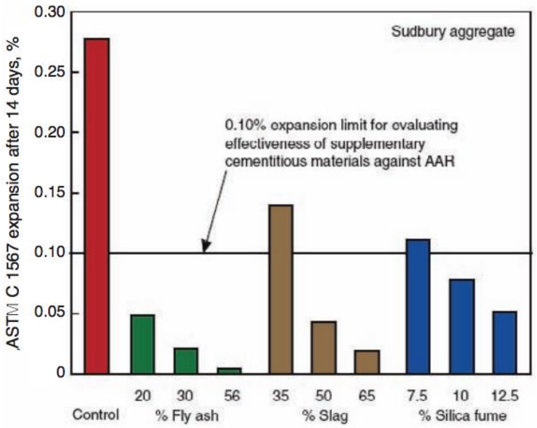
SCM types and screening

- SCMs can be pozzolanic or latent hydraulic
- Pozzolanic SCMs react with calcium hydroxide (CH) and water at high pH to form C-S-H
- Latent hydraulic SCMs react with water to form C-S-H once activated (do not need CH)
- Inert fillers do not react with water or calcium hydroxide; <u>they can show other reactions</u>
- It is **critical** to differentiate pozzolanic, latent hydraulic, and inert materials
  - Used at different replacement levels in concrete; pozzolanic < 25%, latent hydraulic < 40%, inert < 15%
  - Different benefits for concrete strength and durability (ASR)
  - How to screen/measure reactivity?

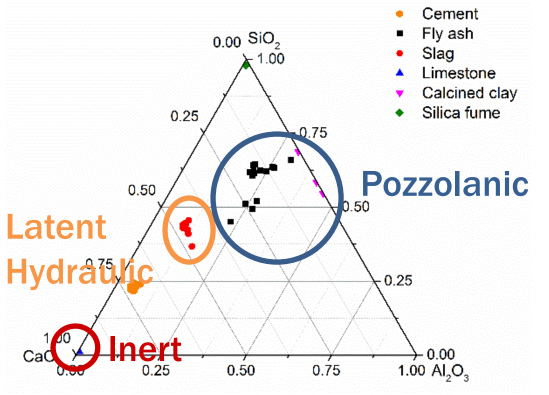


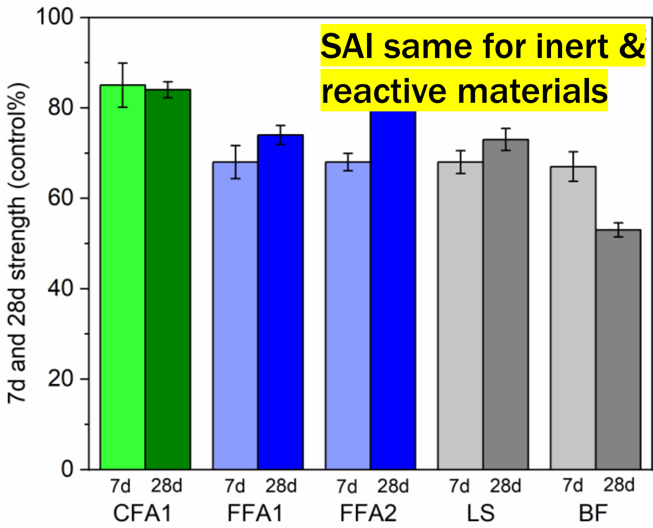


Antoni et al. CCR 2012; Suraneni et al. JMCE 2018; Farny and Kerkhoff, Concrete Technology

SCM reactivity tests

- Could simply using SCM chemistry work?
  - No (Consider silica fume/quartz); amorphous content reqd.
- ASTM C618 uses strength activity index test
  - Test does not work well; most inert fillers pass because age of testing, replacement level, and test limits too low
- Tests based on calcium hydroxide consumption do not accurately quantify reactivity of latent hydraulic SCMs
- Do we need to distinguish pozzolanic and latent hydraulic SCMs?
- Total vs. pozzolanic reactivity and the role of Ca



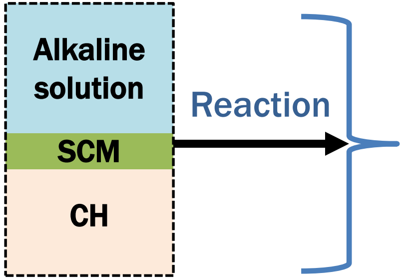


Suraneni et al. ACI MJ 2021; ASTM C311; Pourkhorshidi et al. CCC 2010; Kalina et al. ACI Mater. 2019; Donatello et al. CCC 2010; Kasaniya et al. ACI Mater. 2019; Ramanathan et al. CCC 2020a; Snellings and Scrivener MS 2016; Wang et al. CCC 2021a

SCM reactivity tests

- SCMs react with CH/water at high pH to form C-S-H
  - If adequate amount of reaction occurs, then the material is an SCM; else it is not
- Various aspects of the reaction can be measured in model systems/pastes/mortars
  - Chapelle test, Frattini test, R^3, and modified R^3 tests (model systems)
  - Lime strength test, SAI, BRI, and variants (mortars)
  - Calcium hydroxide, bound water, heat release, strength, bulk resistivity of cement-SCM systems (paste/mortars)
- Ideally
  - Differentiate inert, pozzolanic, latent hydraulic SCMs
  - Estimate strength and durability aspects
  - Rapid, robust, reliable + cheap & simple

Measures of reaction in an SCM model system of varying composition, with or without cement and sand



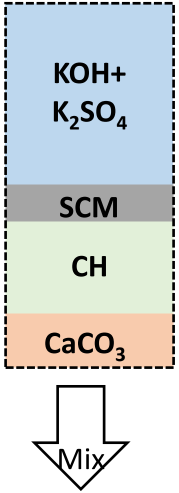
Measure
Dissolution
Heat release
Bound water
CH consumption
Strength
Bulk resistivity

Can vary mixture proportions, solution composition, temperature, pH

Avet et al. CCR 2016; Snellings and Scrivener M&S 2016; Suraneni and Weiss CCC 2017; Li et al. MS 2018; Suraneni et al. CCC 2018; Snellings et al. ACI MJ 2019; Suraneni at al. CCC 2019; Kasaniya et al. ACI MJ 2019; Wang and Suraneni CBM 2019; Glosser et al. ACI MJ 2019; Ramanathan et al. CBM 2019; Palou et al. JTAC 2020; Blotevogel et al. CCR 2020; Ramanathan et al. CCC 2020a and 2020b

R^3 and modified R^3 tests

40 °C

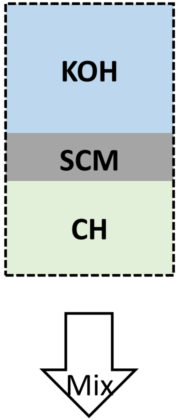


R^3 test

- ✓ CH/SCM = 3:1
- ✓ $CaCO_3$/SCM = 1:2
- ✓ KOH = 0.24 g, K_2SO_4 = 1.20 g, water = 60 g
- ✓ Isothermal calorimetry at 40 °C for 7 days **OR**
- ✓ Oven dry the specimen for bound water

OR

50 °C



Modified R^3 test

- ✓ CH/SCM = 3:1
- ✓ Liquid-to-solid ratio 0.9, 0.5 M KOH (pH 13.5)
- ✓ Isothermal calorimetry at 50 °C for 10 days
- ✓ Thermogravimetric analysis at 10 days for CH consumption and bound water

and

Avet et al. CCR 2016; Suraneni and Weiss CCC 2017

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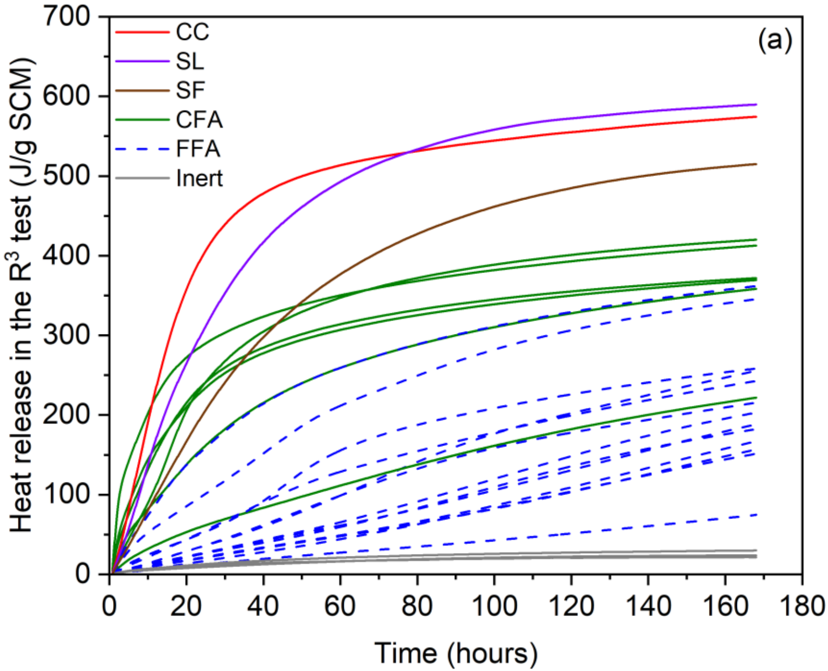
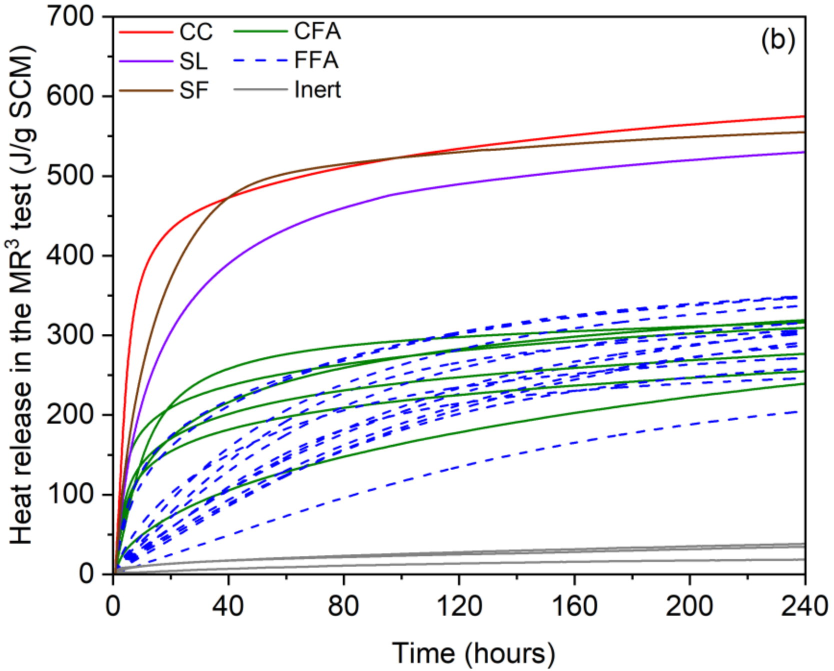
R^3 test heat release (ASTM C1897)

- Inert materials (LS, BF, and Q – heat release < 50 J/ SCM)
- Fly ashes reactive (50 – 400 J/g SCM), clear differences between Class C and Class F fly ashes
- More reactive CC, SL, and SF (500 – 600 J/g SCM)
- **Heat release for Class F fly ashes still increasing at 7 days**
- **Differences between low reactive and slowly reacting materials important**



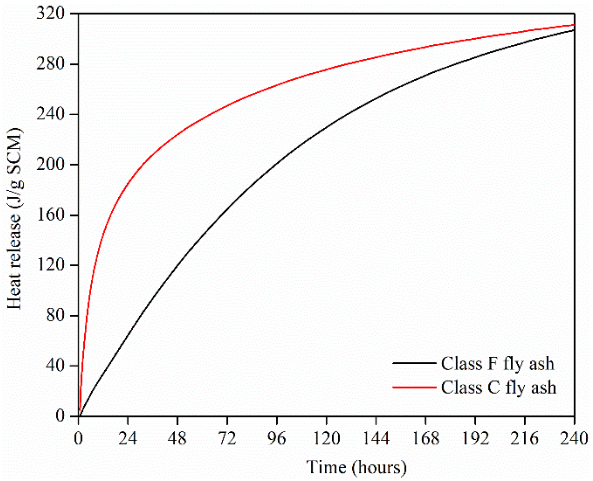
MR^3 test heat release

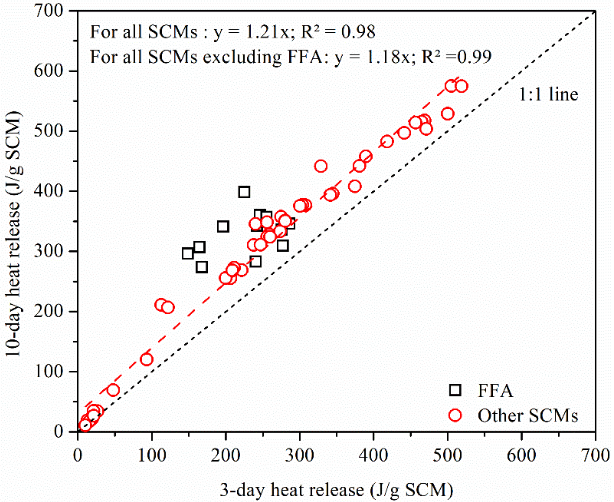
- Inert materials (heat release < 50 J/ SCM)
- Fly ashes reactive (200 – 350 J/g SCM), differences between Class C and Class F fly ashes at 1 day but not at 10 days
- More reactive CC, SL, and SF (500 – 600 J/g SCM)
- All materials plateau at 10 days



Reaction kinetics

- Differences between Class C and Class F fly ashes very clear at early ages (< 3 days) but not at later ages (10 days)
- Power laws fit to heat flow curves and most of the reaction is completed by 3 days





- Test duration can be reduced to 3-days, reducing experimental effort and cost
- Caution needed with Class F fly ashes and slowly reacting SCMs... especially if testing is at 40 °C!

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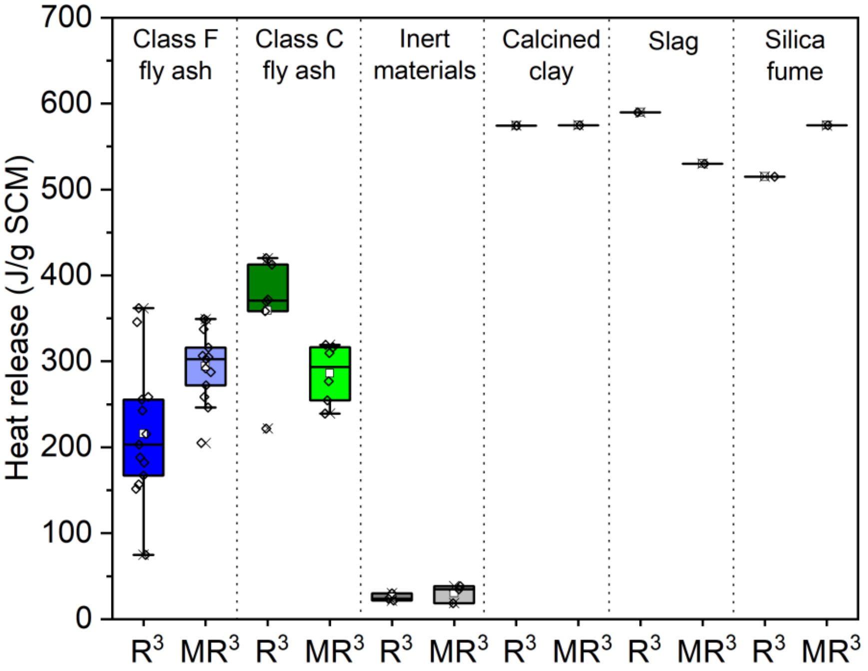
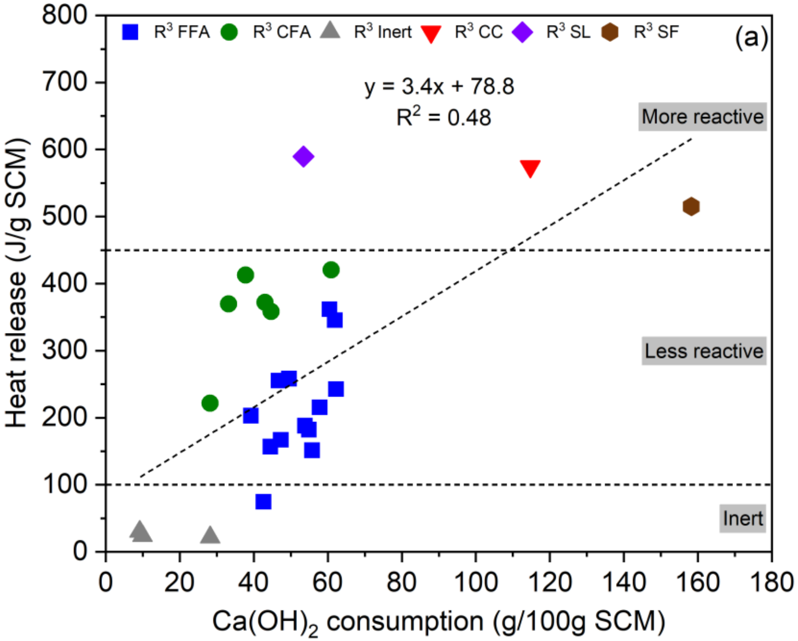
Test comparison

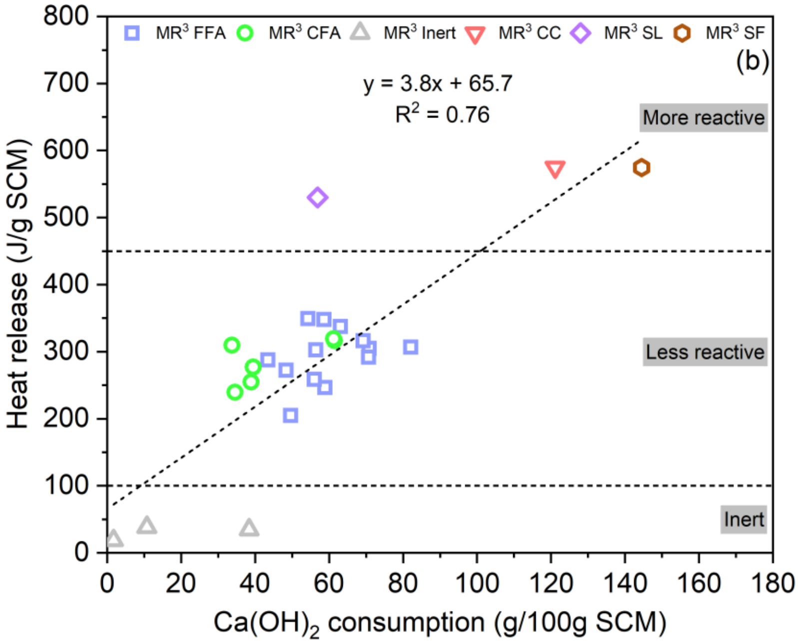
- **All fly ashes reactive**
- Inert materials – low heat release in both tests
- Low C + A materials (Class F and SF) – temperature dominates, Heat ($MR^3 > R^3$)
- High C + A materials (Class C, SL, and CC) – sulfates and carbonates dominate, Heat ($R^3 > MR^3$)
- Do these differences matter?



Heat release & CH consumption

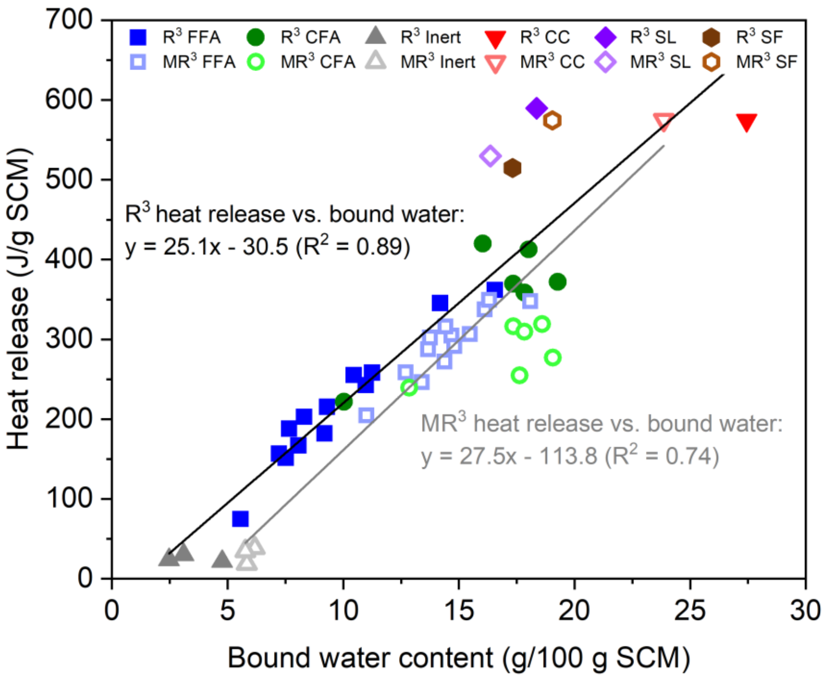
- **Both tests differentiate inert, less reactive, and more reactive materials**
- MR^3 test shows better Heat release – CH consumption correlation than R^3 test due to 'fly ash vertical offset'
- Why - secondary reactions not consuming Ca?





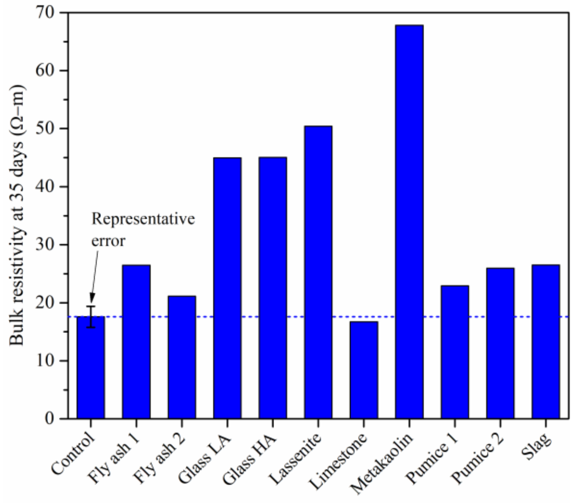
Heat release & bound water

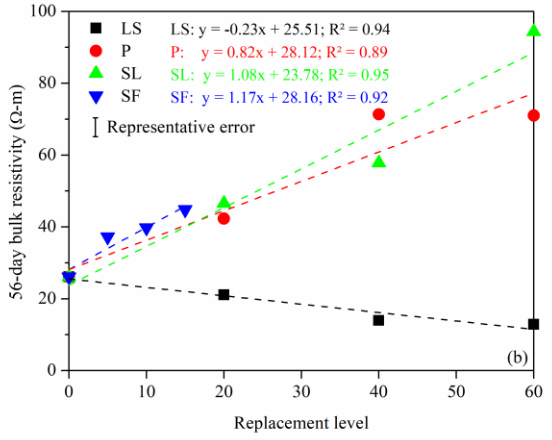
- Positive linear correlations between the heat release and the bound water
- More material reacts → More water is bound → More heat is generated
- R^3 test shows better correlation than the MR^3 test
- Why?
- A furnace could be used to estimate bound water and CH consumption



Bulk resistivity

- Bulk resistivity of cement pastes shows promise in differentiating SCMs and inert fillers
  - Inert fillers reduce resistivity, SCMs increase it
  - Indirect reactivity measure (changes in pore solution, pore structure, alkali binding…)
  - Differences especially apparent at high replacement levels, high temperatures, and later ages
  - Bulk resistivity highly sensitive to reactivity and a durability measure



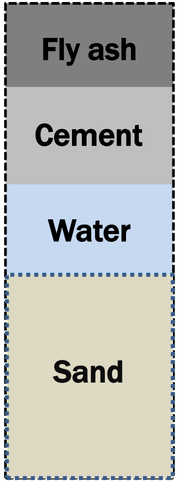


Ramanathan et al. CCC 2020a; Ramanathan et al. CCC 2020b

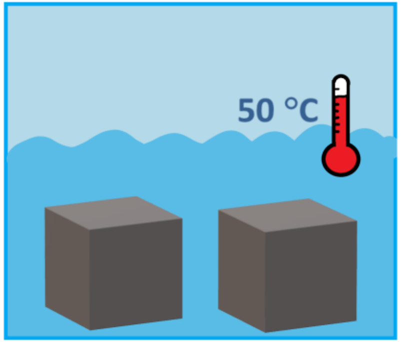
Bulk resistivity index (BRI)

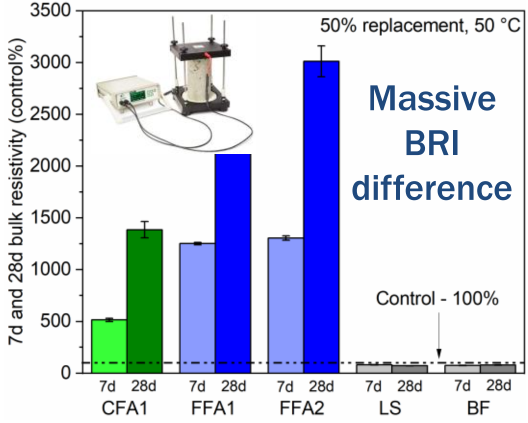
- Considering the flaws of the ASTM C311 strength activity index, a simple fix is to use the **bulk resistivity index** as an alternative

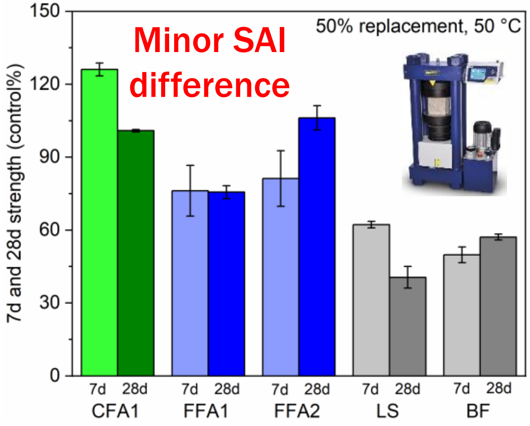
Higher replacement level (50%)



Higher curing temperature (50 °C)



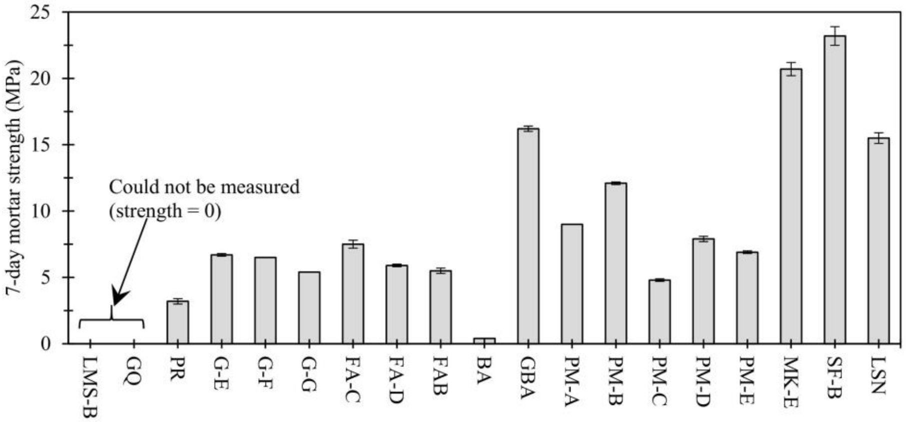




Ramanathan et al. CCC 2020a; Ramanathan et al. CCC 2020b; Wang et al. CCC 2021a

Lime strength test

Test	Lime strength test
Mixture design	CH: SCM 1:1.5, w/b 0.65, s/b 2.5, $CaCO_3$/(SCM + CH + $CaCO_3$) 1:15; K_2SO4/KOH = 5 and 0.3 M K^+ solution
Curing conditions	Mortars stored over water at 23 °C (1 day), in an oven at 40 °C (2 days), and then in water at 40 °C (4 days)
Properties measured	Compressive strength
Reactivity threshold	7-day strength 3 MPa
Classifi-cation	Classes from inert to very high reactivity proposed



- Measure strength of lime pozzolan mortars
- Greater strength, greater reactivity
- Correlations shown to R^3 results and to mortar properties

Kasaniya et al. CCR 2021; Kasaniya et al. CBM 2022

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Standards & specifications

- The R^3 test is already a standard – ASTM C1897
- We could standardize the modified R^3 test but this needs a lot of work and data
- Maybe more valuable to get ASTM C1897 into specifications
- Heat release, bound water, CH consumption are similar enough that any one could be measured and be acceptable
- Especially if combined with chemical composition information
- There is merit in standardizing the bulk resistivity index and lime strength test
- They are 'simpler' than R^3 type tests or at least perceived to be
- Everyone is familiar with a compressive strength test and in principle any lab could run the lime strength test

Poor man's reactivity test

- If you want to qualitatively test SCM reactivity without running any tests...
- Mix 5 grams SCM, 15 grams CH, 18 grams 0.5 M KOH, store at 38/40/50 °C in a sealed container
  - If material rapidly hardens in < 1 day (can't easily deform), material highly reactive
  - If material hardens in < 3 days, material moderately reactive
  - If material hardens in 3 - 14 days, material mildly reactive
  - If material never hardens, inert
- May run a Vicat test on these SCMs and compare with strength/heat

Concluding thoughts

- Measure reactivity, it is very important, especially for emerging/future SCMs
- Ideally, do it according to a standard (R^3 ASTM C1897) but modified R^3, bulk resistivity, lime strength also ok
- Check with threshold level and benchmark reactivity with that of other known materials
- Reactivity gives some idea of strength and durability
- ... but still need to measure paste/mortar/concrete properties

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