Reactivity tests for Supplementary Cementitious Materials (SCMs)
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RILEM TC-267 TRM committee

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Decrease of the availability of traditional SCMs

- New sources or combination of SCMs are considered
- Wide availability of calcined clay with various grades and also limestone

![Graph showing the availability of various SCMs](image)

- Limestone
- Calcined clays
- Rice husk ash
- Silica fume
- Burnt shale
- Natural pozzolana
- Blast furnace slag
- Fly ash
- Cement

Fly ash: significant volumes with low performance

Scrivener K., Gartner E., John V. UNEP report (2016)
Assessment of SCM reactivity

- Usual way: measurement of compressive strength of blended cement
- However, this test is time and material consuming (28 days as reference value)

- Reactivity tests developed to get a quicker indication of the reactivity of SCMs
- Ideally, tests as quick, simple, robust, reproducible and cheap as possible

**RILEM TC 267-TRM** aims to compare existing and innovative reactivity tests to give a recommendation that can be adopted as standard testing method.

- 21 participants
- 11 SCMs
- 10 different techniques
Objectives of the RILEM TC 267-TRM

Phase I work:
- Comparison of existing and novel methods
- Test on a wide range of SCMs
- Correlation with reference mortar strength
- Selection of the most accurate tests

Phase II: Test robustness of protocols
- Identification of key parameters
- Improve the protocols
### Materials and Methods

<table>
<thead>
<tr>
<th>SCMs</th>
<th>Test</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 calcined clays</td>
<td>Chapelle test or modified version</td>
<td>NF P18-513</td>
</tr>
<tr>
<td>2 slags</td>
<td>Frattini test</td>
<td>EN 196-5</td>
</tr>
<tr>
<td>2 calcareous fly ashes</td>
<td>Reactive silica</td>
<td>EN 197-1 / EN 196-2</td>
</tr>
<tr>
<td>2 siliceous fly ashes</td>
<td>Lime reactivity test</td>
<td>IS 1727</td>
</tr>
<tr>
<td>Natural pozzolana</td>
<td>R³ test</td>
<td>-</td>
</tr>
<tr>
<td>Quartz as inert</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **Measures the reactivity of SCM based on CH characterization**
- **Compressive strength measurement of SCM:CH binary blends**
Monitoring reactivity of SCMs: R³ test

- Rapid, Relevant and Reliable (R³)
- Focus on SCM reaction only
  - Adjustment of sulfate and alkali content to reproduce the reaction environment of hydrating blended cements

Two ways of measuring the reactivity

<table>
<thead>
<tr>
<th>Components</th>
<th>Mass (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCM</td>
<td>11.11</td>
</tr>
<tr>
<td>Portlandite</td>
<td>33.33</td>
</tr>
<tr>
<td>Deionized Water</td>
<td>60</td>
</tr>
<tr>
<td>KOH</td>
<td>0.24</td>
</tr>
<tr>
<td>K₂SO₄</td>
<td>1.20</td>
</tr>
<tr>
<td>Calcite</td>
<td>5.56</td>
</tr>
</tbody>
</table>

Isothermal calorimetry at 40°C Heat release 7d

Oven thermal treatment at 350°C Bound water 7d
Adjustment of portlandite to SCM ratio

- Plateau reached for 1/3, 1/2 and 1/1
- In order not to run out of portlandite, 3/1 was chosen

Avet et al. (2016)
Determination of bound water

- Mass evolution after thermal treatment at 350°C for 2 hours
- Only requires a balance and an oven

![Graph showing mass evolution and temperature over time.](image-url)
Results: Strength test as reference (30% substitution)

- 6 cements used in 6 different labs
- Significant differences, even though all cements used for the blends are CEM I 42.5 R
Correlation between reactivity tests and strength

![Graphs showing correlation between reactivity tests and strength](image)
Phase I: Most promising results obtained with the R³ test

- Frattini and Chapelle tests give poor correlation to strength, with very low interlab reproducibility.

- Improvement of Frattini by excluding slags

- R³ tests using calorimetry and bound water give high correlation to strength and are the most reproducible

- R³ deeper investigated in phase II for improving the protocols and the robustness

Li et al (2018)
Reactivity tests for supplementary cementitious materials: RILEM TC 267-TRM phase 1

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Phase I work:
- Comparison of existing of novel methods
- Test on a wide range of SCMs
- Correlation with reference mortar strength

Phase II: Test robustness of protocols
- Identification of key parameters
- Improve the protocols
Investigation of the robustness and reproducibility of $R^3$

- $R^3$ Heat release
  - Premixing and mixing conditions
  - Mix design composition
  - Water bath

- $R^3$ Bound water
  - Drying procedure
Premix and mix conditions

![Graph showing the impact of premix and mix conditions on heat generation over time. The graph includes lines for different mixing methods (MH, MM, TM) and time points (0.5 d, 1 d, 3 d, 7 d).](image)

- **Part 1:** Premix
- **Manual premix**
- **Hand paste mixing**
- **Mechanical paste mixing**
- **Calorimetry**

Legend:
- **CC2**: Control condition 2
- **S8**: Sample 8
- **SFA_R**: Sample with rapid fermentation

Heat generated (J/g SCM) vs Time (Days)
Mix composition change

(5 participants)

Accumulative heat of CC2 (average)

Accumulative heat of S8 (average)
Mix composition change

(5 inputs)

<table>
<thead>
<tr>
<th></th>
<th>X15</th>
<th>X25</th>
<th>X40</th>
<th>W10</th>
<th>W13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mortar_28</td>
<td>1.00</td>
<td>0.98</td>
<td>0.88</td>
<td>0.97</td>
<td>1.00</td>
</tr>
<tr>
<td>Mortar_90</td>
<td>0.49</td>
<td>0.38</td>
<td>0.18</td>
<td>0.35</td>
<td>0.56</td>
</tr>
<tr>
<td>CV (%)</td>
<td>18.7</td>
<td>5.6</td>
<td>6.4</td>
<td>6.9</td>
<td>8.7</td>
</tr>
</tbody>
</table>

Initial mix
Water bath during casting

(5 participants)

Accumulative heat (average)
Investigation of the robustness and reproducibility of $R^3$

- $R^3$ Heat release
  - Premixing and mixing conditions
  - Mix design composition
  - Water bath

- $R^3$ Bound water
  - Drying procedure
Drying step investigation

Small pieces cut and stored at 105°C

Pieces stored at 350°C for 2 hours

Bound water

Mass Stabilization

Temperature (°C)

Mass (g)

Time (days)
Bound water – different drying procedures

(7 participants)
Bound water – different drying procedures
(7 participants)

Correlation to relative strength

<table>
<thead>
<tr>
<th>R2</th>
<th>Boundwater</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BW_50</td>
</tr>
<tr>
<td>Mortar_28</td>
<td>0.92</td>
</tr>
<tr>
<td>Mortar_90</td>
<td>0.24</td>
</tr>
<tr>
<td>CV (%)</td>
<td>22.7</td>
</tr>
</tbody>
</table>
Bound water – different drying procedures

![Graph showing bound water (g/100 g paste) for different drying procedures and times. The graph includes data for CC2, SFA_R, Q, and S8 at 40°C, 50°C, and 105°C. The box plots indicate the variability in bound water with different drying times and temperatures.]

- **Phase2+ 40°C**
- **Phase2+ 50°C**
- **Phase2+ 105°C**

The box plots for 48 hours, 24 hours, and 3 hours at each temperature show the distribution of bound water values, indicating the variability and central tendency of the data.
Conclusion on phase II

- Finalization of the protocols
  - Heat of hydration
    - Premix or mixing did not show significant impact on the results
    - Recommended to use mechanical mixing if available
    - 25% of SCM in the mix design is a good compromise
    - 1.2 water to binder ratio works well
    - Water bath does not really help
  
- Bound water
  - Drying step at 40°C, simpler and better reproducibility

- Standard in preparation for using in phase III
Phase III

- Validation across wide range of SCMs

- Definition of scope of test method – boundary conditions
  - Conventional SCMs and material currently falling outside of standards
  - Dependence on clinker replacement ratios
  - Impact of temperature
  - Water to binder ratio
Participation in the RILEM TC 267-TRM reactivity test

The RILEM TC-TRM “Tests for Reactivity of Supplementary Cementitious Materials” concluded that two main methods (R3 calorimeter and oven technique) are the most promising for evaluating the reactivity of SCMs. The next step is to apply these two methods to a broader and a more substantial number of new and more conventional SCMs.

If you want your material to be tested, requirements are:

- Fill the online form: Deadline: 31st December 2018
- Quantity of SCM: 50 kg of dry ground homogenized SCMs to be sent to EPFL (Switzerland) before the end of April 2019
- Fineness of SCM: $d_{50} < 20 \mu m$

What we offer:

- SCM characterization
- Testing of reactivity through mortar test and R3
- Report on the assessment of reactivity
- Cost: 500 euros for participation + shipping cost

Form for SCM testing RILEM TC 267-TRM

Deadline: 31st December 2018

Name: First name:
Address:
Zip code:
City:
Country:

Description of your material:

Preparation:

Rough composition / presence of hazardous materials:

Our committee will screen all the application forms and will contact you for the acceptance of your material by the end of January 2019.
3rd workshop at EPFL, Switzerland, April 2017

4th meeting in Chennai, India, September 2017

5th meeting in Leuven, Belgium, April 2018

Thank you for your attention

1. https://www.rilem.net/groupe/267-trm-tests-for-reactivity-of-supplementary-cementitious-materials-
Correlation between reactivity tests and strength
Different calorimeter (U. Toronto - Calmetrix)

Accumulative heat (average)
Selection of the cement – PC strength and relative strength for Q
Selection of the cement – relative strength CC2 and S8

- Calcined clay (CC2)
- Slag (S8)