Reactivity tests for supplementary cementitious materials: RILEM TC 267-TRM phase 1

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Abstract:

A primary aim of RILEM TC 267-TRM: "Tests for Reactivity of Supplementary Cementitious Materials (SCMs)" is to compare and evaluate the performance of conventional and novel SCM reactivity test methods across a wide range of SCMs. To this purpose, a round robin campaign was organized to investigate 10 different tests for reactivity and 11 SCMs covering the main classes of materials in use, such as granulated blast furnace slag, fly ash, natural pozzolan and calcined clays. The methods were evaluated based on the correlation to the 28 days relative compressive strength of standard mortar bars containing 30% of SCM as cement replacement and the interlaboratory reproducibility of the test results.

It was found that only a few test methods showed acceptable correlation to the 28 days relative strength over the whole range of SCMs. The methods that showed the best reproducibility and gave good correlations used the R^3 model system of the SCM and $Ca(OH)_2$, supplemented with alkali sulfate/carbonate. The use of this simplified model system isolates the reaction of the SCM and the reactivity can be easily quantified from the heat release or bound water content. Later age (90 d) strength results also correlated well with the results of the IS 1727 (Indian standard) reactivity test, an accelerated strength test using an SCM/$Ca(OH)_2$-based model system. The currently standardized tests didn't show acceptable correlations across all SCMs, although they performed better when latently hydraulic (slag) materials were excluded. However, the Frattini test, Chapelle and modified Chapelle test showed poor interlaboratory reproducibility, demonstrating experimental difficulties. The TC 267-TRM will pursue the development of test protocols based on the R^3 model systems. Acceleration and improvement of the reproducibility of the IS 1727 test will be attempted as well.

Keywords: supplementary cementitious materials, reactivity test, heat release, bound water, compressive strength

1 Introduction

The use of supplementary cementitious materials (SCMs) as a partial replacement for clinker in blended cements or concrete is becoming increasingly widespread. In addition, the availability of traditionally used SCMs (e.g. blast furnace slag and fly ash) is decreasing and a wider range of materials and combination are being considered as SCMs. The first criterion for such replacements is the contribution they make to the development of mechanical properties so there is great interest both in testing this directly and in the development of tests which give a rapid assessment of this reactivity. RILEM TC 267-TRM (Tests for Reactivity of Supplementary Cementitious Materials) was established to evaluate the existing reactivity tests and develop a pre-normative recommendation for rapid SCM reactivity tests that can be adopted as standard testing methods. Ideally test methods should supply results more rapidly than the standard compressive strength testing regimes, they should be straightforward and robust to execute and should not require expensive equipment or advanced training of practitioners.

The current standardized methods for SCM or pozzolanic reactivity test are 1) the Chapelle test [1] or a modified version of it (NF P18-513) [2], 2) the Frattini pozzolanicity test (EN 196-5) [3], and 3) the determination of reactive silica (EN 196-2:2013). An Indian standard (IS 1727-1967) – locally known as the lime reactivity test - is also in use. Both the (modified) Chapelle [4,2,1] and Frattini test methods [4,3] measure the reactivity of the SCM with $Ca(OH)_2$, either by titrating the amount of $Ca(OH)_2$ remaining in a dilute suspension or by evaluating the saturation degree of solution towards $Ca(OH)_2$, respectively. Both tests intend to test pozzolanic reactivity (with portlandite) and were not intended to work for latent-hydraulic, Ca-rich SCM such as blast furnace slags. The Chapelle test takes less than 1 day to carry out, the Frattini test at least 8 days, and for less reactive SCMs up to 15 days. The IS 1727 test measures the compressive strength of a portlandite ($Ca(OH)_2$) and SCM binary mix cured initially at 27°C and then at 50 °C until 10 days after casting. Previous work, indicated that these standard reactivity testing methods for supplementary cementitious materials have shortcomings [4], particularly in terms of correlation to strength development of cements, test duration and reproducibility.

There has been much research on the mechanism of reaction of SCMs in blended systems, which has benefited from advances in analytical methods and thermodynamic modelling [5,6]. In contrast, few advances have been made regarding reactivity test methods.

More recently, the spread of new or improved experimental techniques such as isothermal conduction calorimetry has inspired new research into the topic [7,8]. The so called "R^3" test was developed initially to test the pozzolanic activity of calcined clays. R^3 stands for rapid, reproducible and relevant: The aim is to have a method which can give results correlating to strength in standard mortars (*relevant*) in a much shorter time (*rapid*) and which is relatively simple to carry out giving *reproducible* results. This method tries to better simulate the conditions occurring in a blended cement by the addition of small amounts of sulfate and alkali to an SCM portlandite mixture [8]. This test was accelerated by measuring the reaction at 40 °C either by the heat release in isothermal calorimetry continuously up to 7 days, or bound water between 110 °C and 40 °C after 7 days of curing [8]. For calcined clay a very good correlation was found between the amount of reaction at 1 day at 40 °C and the 28 day strength in standard mortar bars.

This paper reports on the round robin study which was phase 1 of the committee work. The objective was to look at the performance of a range of methods proposed to measure reactivity across a wide range of SCMs. The tests methods were selected according to the experience of the committee members and an overview of SCM reactivity tests [4]. Two categories of test methods were defined: the existing standard methods and the R^3 model system tests (non-standard). For the R^3 system, measurements of portlandite consumption using thermogravimetric analysis (TG) [9] and the chemical shrinkage [10] were included in addition to the calorimetry and bound water methods.

The reactivity test results on a selected range of SCMs were compared to a benchmark - the compressive strength results of cement mortar bars (EN 196-1) - in which 30 wt.% of the Portland cement was replaced by SCM. The interlaboratory reproducibility of the test methods was assessed. A selection of test methods which seem to be giving best results for further testing and optimization was made for the phase 2 work of this committee.

2 Experimental

2.1 Participants and work plan

In total, there were 21 participants (see Table 1), who were free to choose which methods to test. The summary of the number of participants for each test is shown in Table 2.

Table 1 Summary of the participants

Continent	**Europe**		**North America**	**Asia**
Participants	Empa	RWTH Aachen	U. Laval	IIT Delhi
	EPFL	TU Delft	U. Texas Austin	IIT Madras
	LafargeHolcim	Università degli Studi della Basilicata	U. Toronto	Sinoma
	HeidelbergCement Technology Center	U. Gent		
	INSA Rennes	U. Sheffield		
	INSA Toulouse	VITO		
	KU Leuven	ZAG		
	NTNU			
Subtotal	15		3	3
Total	**21**			

Notes: U. = University,

Table 2 Summary of the test planning.

	Test	***Total participants***
Standard method	Mortar test: EN 196-1	6
	Frattini test: EN 196-5	5
	Chapelle test: standard	4
	Modified Chapelle test: NF P18-513	5
	IS 1727 (Indian standard)	2
	Reactive silica: EN 197-1/EN 196-2	1
R^3 model	Calorimetry	13
	Bound water	13
	Chemical shrinkage	5
	Portlandite consumption	7

2.2 SCMs

In this phase 1, the aim was to look at a wide range of SCMs, including those most commonly used. Eleven materials, were selected:

- 2 calcined clays (labelled as *CC1* and *CC2*)
- 2 ground granulated blast-furnace slags (labelled as *S1* and *S8*)
- 2 calcareous fly ashes from coal combustion (labelled as *CFA_P* and *CFA_S*)
- 3 siliceous fly ashes from coal combustion (labelled as *SFA_E*, *SFA_I* and *SFA_R*)
- 1 natural pozzolan (labelled as *Po*)
- quartz (labelled as *Q*) as a reference for an inert material

The Supplementary Material gives the chemical composition (measured by X-ray fluorescence analysis); origin and the physical properties of the SCMs (Blaine fineness, density measured according to ASTM C188-09 using isopropanol instead of kerosene [11]; particle size distribution (PSD) measured using Malvern laser diffraction using isopropanol); mineralogical compositions of the materials obtained by X-ray powder diffraction (XRD) with Rietveld analysis. For SCMs, the external standard method was used to determine the amorphous content (details on the XRD experiments and Rietveld analysis are given in supplementary material).

2.3 Benchmark testing

It was decided to use a classic strength test as a benchmark for the reactivity tests. The level of replacement of the SCMs was chosen as 30% to give good sensitivity to the contribution of the SCMs.

Six participants carried out the mortar strength tests according to EN 196-1 using local Portland cements (in total 6 different cements were used) of type CEM I 42.5 N/R or similar. The characteristics of the cements used for the mortar tests are given in the Supplementary Material.

The mortar tests were carried out according to EN 196-1. 30% by mass replacement of cement by SCMs was used, an adjustment of gypsum content (similar to Antoni et. al. [12]) was applied and superplasticizer (PCE type) was introduced for calcined clays to control the reaction of the Al_2O_3 in the SCMs and the workability of the mortar, respectively. The compressive strength was measured at 2, 7, 28 and 90 days.

It was not possible to average the absolute strengths for the different cements as 6 local CEM I 42.5N/R cements were used. So, the relative compressive strength $R_{SCM,relative}$ (%) was used for the correlation analysis:

$$R_{SCM,relative} = \frac{R_{SCM} - R_{PC}}{R_{PC}} \times 100 \qquad \text{Eq. (1),}$$

where R_{SCM} and R_{PC} are the absolute strength in MPa for the SCM blended cement and the pure PC from the same source, respectively. The $R_{SCM,relative}$ was calculated for each cement and then averaged. The strengths relative to the quartz references were also calculated.

2.4 Methods

Detailed protocols for each of the methods are given in the Supplementary Material.

2.5 Standard SCM reactivity tests

The standardised methods used were:

1) Chapelle test or a modified version of it (NF P18-513)

The Chapelle test [1] assesses the consumption of calcium hydroxide by a test material in a dilute heated suspension as a measure of pozzolanic activity. 1 g of SCM is reacted with 1 or 2 g of $Ca(OH)_2$ (Chapelle or modified Chapelle test, resp.) in 200 ml of water at 90-100 °C for 16 h. The non-reacted lime is then analyzed and the result expressed in mg $Ca(OH)_2$ fixed by the SCM.

2) Frattini or pozzolanicity test (EN 196-5)

The Frattini test evaluates portlandite saturation in a supernatant solution of a hydrated slurry of Portland cement and a pozzolan test material by measuring the OH^- and Ca^{2+} concentrations. The test consists of mixing a blend of Portland cement (CEM I) and SCM with distilled water at a water to solid ratio of 5. The interpretation of the Frattini test results was made according to Donatello et. al.[13] and Snellings et al.[4], which calculates the vertical distance of data points from the lime solubility curve.

3) The determination of reactive silica (EN 196-2 and EN 197-1)

Reactive silica is defined according to EN 197-1 as that fraction of the SiO_2 which is soluble after treatment with HCl and a boiling KOH solution. The measurement procedure is established in EN 196-2.

4) The Indian test method for pozzolanic materials (IS 1727 - 1967)

In this method, a volume based mix design is used to keep the same volume of the binder in each mix. A 1:2:6 portlandite : pozzolan : sand ratio is used and the w/b ratio is adapted to keep the mortar flow fixed. The mortars are cast and kept in RH saturated conditions and at 27 °C until 2 days, after which the samples are demoulded and further cured at 90-100% RH and 50 °C. The compressive strength of the mortar cubes is measured after 10 days of curing. The strength data are taken as indication of the reactivity of the pozzolan.

2.6 R^3 test

The basic principle of the R^3 test is to use a simplified model system to separately measure the reaction of an SCM. This is to avoid interference and overlap with the clinker hydration reactions that occur in a blended cement system. Moreover, the use of lab-grade chemicals instead of local Portland cements avoids much material related variability. The two main components of the R^3 model system are the SCM and $Ca(OH)_2$. The mix design of the R^3 model paste, shown in Table 3, was based on Avet et. al. [8].

Table 3 R^3 model mix design

Components	SCM	Portlandite[(a)]	Deionized Water	KOH[(b)]	K_2SO_4[(b)]	Calcite[(c)]
Mass (g)	11.11	33.33	60.00	0.24	1.20	5.56

Notes: (a) Lab-grade, less than 5 wt.% $CaCO_3$ present

(b) Lab-grade

(c) Lab-grade, d_{50} 5-15 µm.

The R^3 pastes were used for the bound water, isothermal calorimetry, portlandite consumption using TG and the chemical shrinkage tests.

R^3 bound water

The R^3 pastes were cured in sealed plastic containers at 40 °C for 7 days. The hydrated samples were crushed and dried in an oven at 105 °C until reaching constant weight. The dried samples were heated at 350 °C for 2 hours and the bound water (for hydrates, excluding portlandite) was calculated from the weight difference.

R^3 portlandite consumption

The R^3 pastes were cured in a sealed container at 40 °C for 7 days. The hydration of the samples was stopped by solvent exchange according to [14,15]. The dried samples were analyzed by thermogravimetry. 50 mg of sample was introduced in the crucible which was heated from 30 °C to 950 °C at 10 °C/min. A protective nitrogen atmosphere at a flow rate of 50 mL/min was used. The portlandite content was determined using the tangent method described by Lothenbach et al. [16] and the portlandite consumption calculated by difference to the initial content.

R^3 calorimetry test

Isothermal conduction calorimetry at 40 ^oC was carried out to measure the heat release during hydration of the R^3 systems. The heat release was recorded until 7 days.

R^3 chemical shrinkage

Chemical shrinkage was measured using a modified protocol based on the ASTM C1608-12 and Geiker [17]. 4-6 replicate samples were used for all measurements. The fresh R^3 paste was added into the test vial (weight m_{vial}) up to ~3 cm (half to two thirds of the container's capacity). De-aerated water at 40^{o}C was carefully added on top avoiding mixing with the paste to completely fill the vial. The sealed samples were placed in a water bath at 40 ^{o}C and the volume changes were recorded for 14 days to calculate the chemical shrinkage.

2.7 Data treatment

The inputs from different participants for the same test were averaged, and the standard deviation (σ) on the average of the test results was calculated.

The coefficient of variation (CV, in %) was used to estimate the reproducibility of a test between laboratories: the smaller the CV, the higher the reproducibility. For the calculation of the CV, the difference between the averages of the SCM and the quartz results were used in the denominator. This way the quartz acts as the reference and comparison of the CV amongst samples and techniques is possible.

$$CV_i = \frac{\sigma_i}{\bar{x}_i - \bar{x}_Q} \times 100 \qquad \text{Eq. (2),}$$

where the σ_i and $\bar{x}_i$ are the standard deviation and the mean of the input of a test method from all the laboratories for a specific SCM, respectively. $\bar{x}_Q$ is the mean of the input from all laboratories for quartz for the test method. The mean CV of all the SCMs for a specific testing method was used to assess reproducibility of the method.

Linear fitting of the data from the test methods to relative strength was used for all the SCMs tested. The regression coefficient, R^2, of the linear fitting was taken as the indicator of quality of correlation between the relative strength and the respective test method.

3 Results

The original data are reported in the Supplementary Material. The following sections present an overview of the processed results.

3.1 Compressive strength benchmark test

The strength development of the cement reference samples showed significant differences both at early (2 and 7 days) and late ages (28 and 90 days), even though the cements used for the mortar test were all CEM I 42.5N/R (see supplementary material). These differences were enlarged when the cements were blended with the SCMs. Even when the results were expressed relative to the reference cement, there were still large differences for the results from different laboratories.

The average of the relative strength at 28 days was retained as the critical measure for comparison. For early and later strength, the average of the relative strength at 7 days and 90 days were regarded as the indicators, respectively. The relative strength based rankings of the 10 SCMs and the quartz are shown in Figure 1. The strength relative to the quartz reference were also calculated Figure 1 (d). These show the same ranking (Figure 1 (b) and (d)).

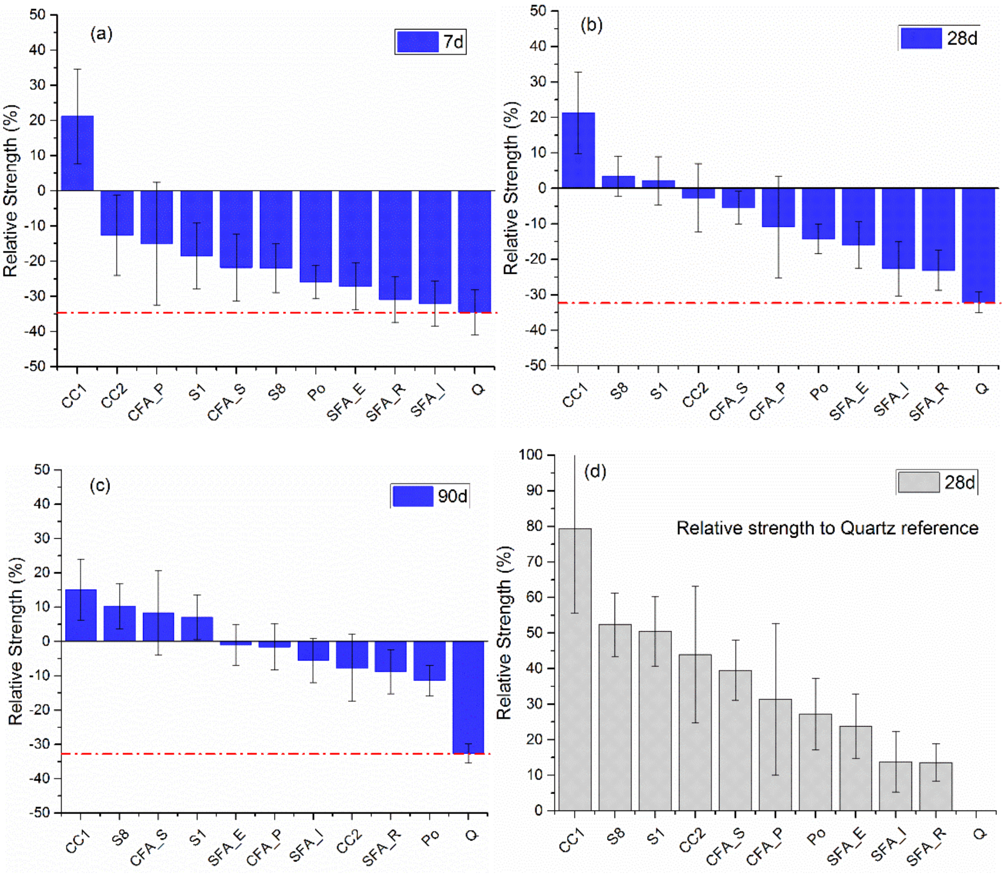

Figure 1 Relative strengths of the SCM blended cement mortar bars, (a), (b) and (c) are relative strengths compared to the PC reference, (d) shows relative strength compared to the quartz (Q) as inert reference

3.2 Correlation analysis of reactivity test results

The global average and the standard deviation of the output for each reactivity test are shown in Figure 2 and Figure 3. Characteristic heat release and chemical shrinkage values at 0.5, 1, 3 and 7 days (and 14 days only for chemical shrinkage) were used for the correlation analysis (the 3 and 7 days values are shown in the Supplementary Material) for continuous measurements such as R^3 calorimetry and chemical shrinkage.

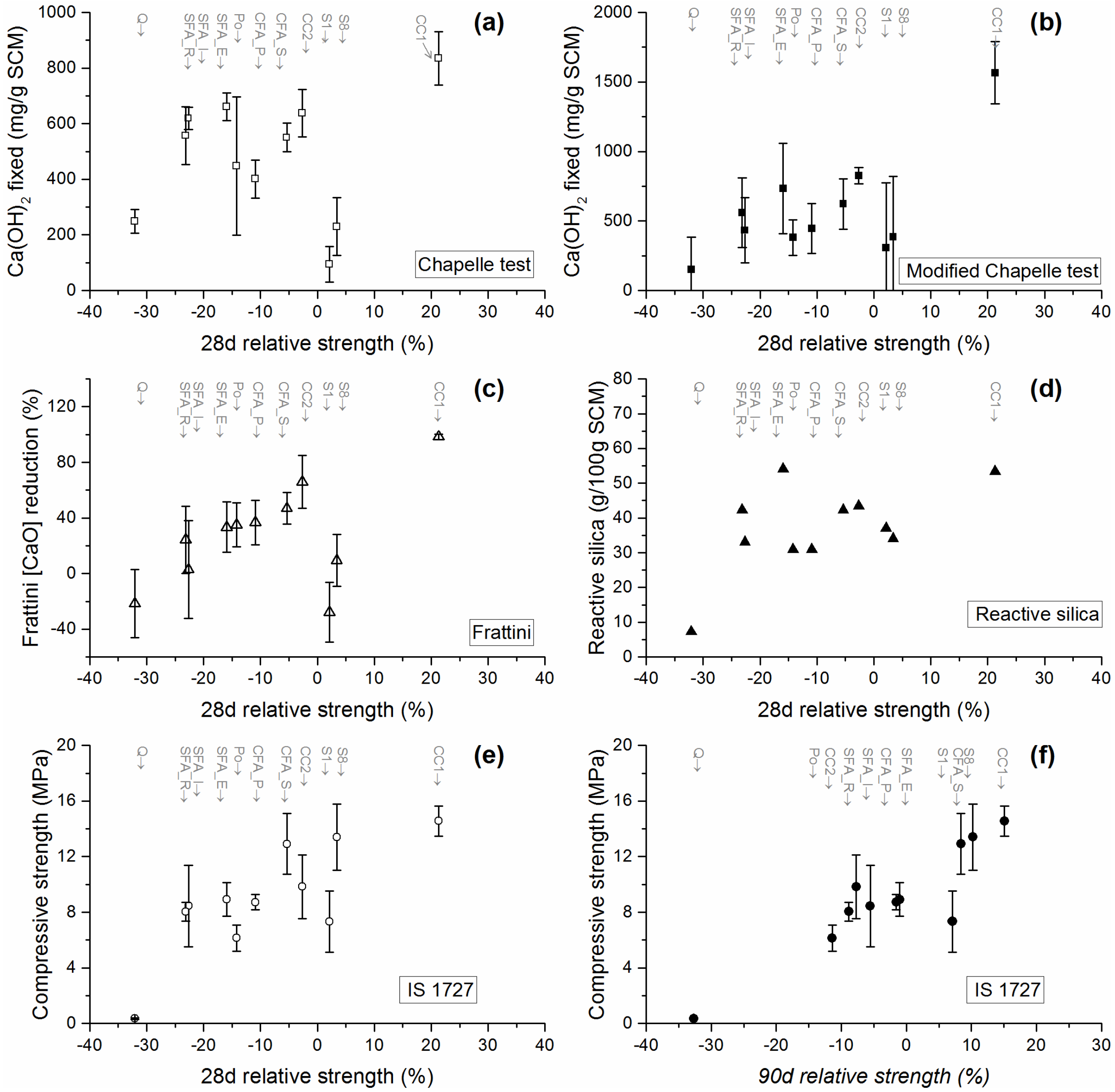

Figure 2 Plots of the standard testing methods against relative strength; the SCMs corresponding to the points are labelled on top of the plot. (a) Chapelle test, (b) modified Chapelle test, (c) Frattini test, (d) Reactive silica, no error bar because there is only 1 input, (e) IS 1727 and (f) IS 1727 (vs. 90 days relative strength). Average values are shown by symbols, the error bars represent 1σ.

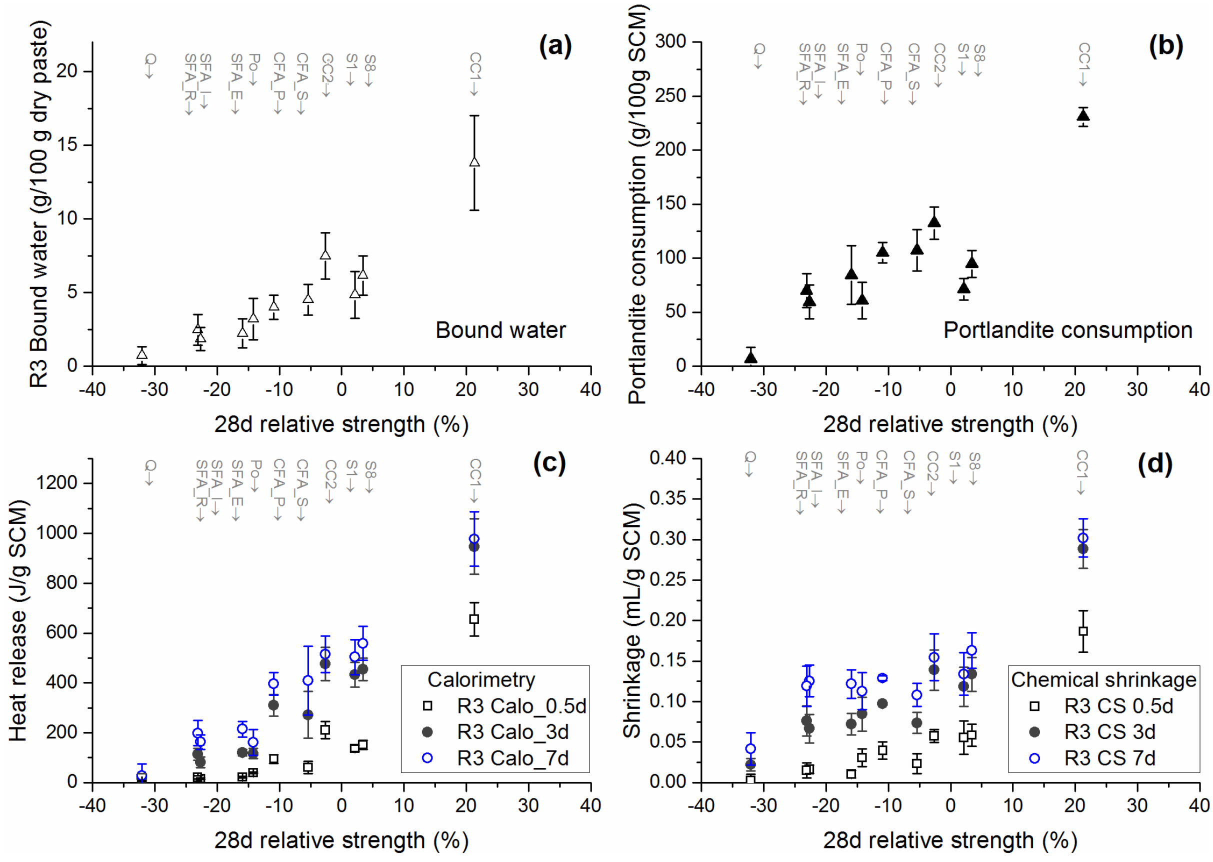

Figure 3 .Plots of the R^3 model test methods to 28 days relative strength, the SCMs corresponding to the points are labelled on top of the plot. (a) Bound water test, (b) Portlandite consumption, (c) Cumulative heat release for 0.5, 3 and 7 days and (d) Chemical shrinkage at 0.5, 3 and 7 days. Average values are shown by symbols, the error bars represent 1σ.

The R^2 values of the linear fitting of the reactivity test to the relative strength using all the SCMs (including the quartz) are summarized in Table 4[2]. Here we considered an R^2 of more than 0.85 as the criterion for acceptance in terms of correlation.

Table 4 .R^2 index of linear correlation of the reactivity test results to the relative strength at 7, 28 and 90 days for all SCMs tested.

Relative strength at	Standard method					R3 model										
	Chapelle	Modified Chapelle	IS 1727	Frattini	Reactive silica	Bound water	CH consum.	Calorimetry (heat released)				Chemical shrinkage				
				[CaO] reduction				0.5d	1d	3d	7d	0.5d	1d	3d	7d	14d
7 days	0.20	0.74	0.39	0.54	0.27	**0.93**	**0.89**	**0.95**	**0.95**	**0.90**	**0.86**	**0.93**	**0.94**	**0.92**	**0.87**	0.72
28 days	0.03	0.46	0.62	0.31	0.33	**0.86**	0.74	0.72	0.80	**0.91**	**0.94**	0.77	0.76	0.80	0.75	0.55
90 days	0.04	0.29	0.82	0.17	0.52	0.43	0.51	0.31	0.36	0.50	0.63	0.36	0.33	0.41	0.49	0.47

3.3 Interlaboratory reproducibility

The coefficient of variation (CV defined in Eq. (8)) was used to indicate the reproducibility of the reactivity test methods (see Figure 4). As there was only one participant for the reactive silica test, the CV was not available

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[2] The R^2 values of the linear correlation of the relative strength to the quartz reference strength are given in the Supplementary Material

for this test. The heat release and chemical shrinkage values at 3 and 7 days were used to evaluate the reproducibility for these continuous tests.

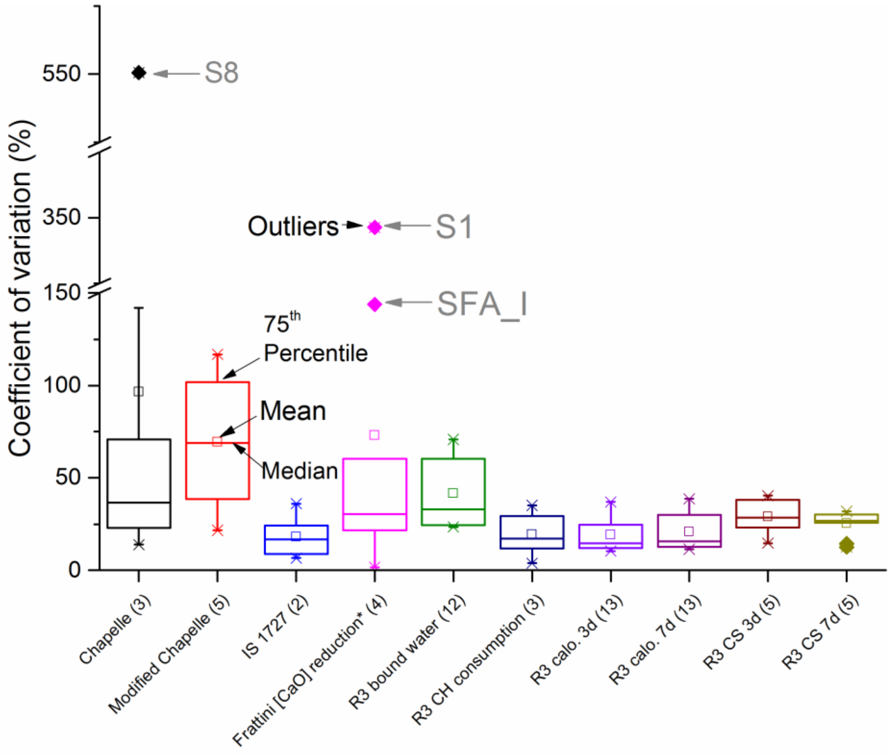

Figure 4 Box chart for coefficient of variation (CV) for different methods, numbers in the bracket along x-axis refer to the number of participants. R^3 CH consumption refers to portlandite consumption for R^3 model test; R^3 calo. 3d and R^3 CS 3d refers to calorimetry heat release and chemcial shrinkage for R^3 model.

4 Discussion: Evaluation of the methods

The test methods were evaluated based on the correlation to the benchmark (relevance) and the interlaboratory reproducibility (reliability). Other factors such as test duration, complexity and cost of equipment also need to be taken into consideration. Figure 5 shows the CV against the R^2 value for the correlation to the 28 days relative strength. An ideal test should be located as close as possible to 1.0 on the R^2 scale while showing the lowest CV in Figure 5. The dotted blue line corresponds to R^2 = 0.85. The results are summarized and compared to the other factors for each reactivity method in Table 5. In the following sections the results for the reactivity test methods are discussed one by one.

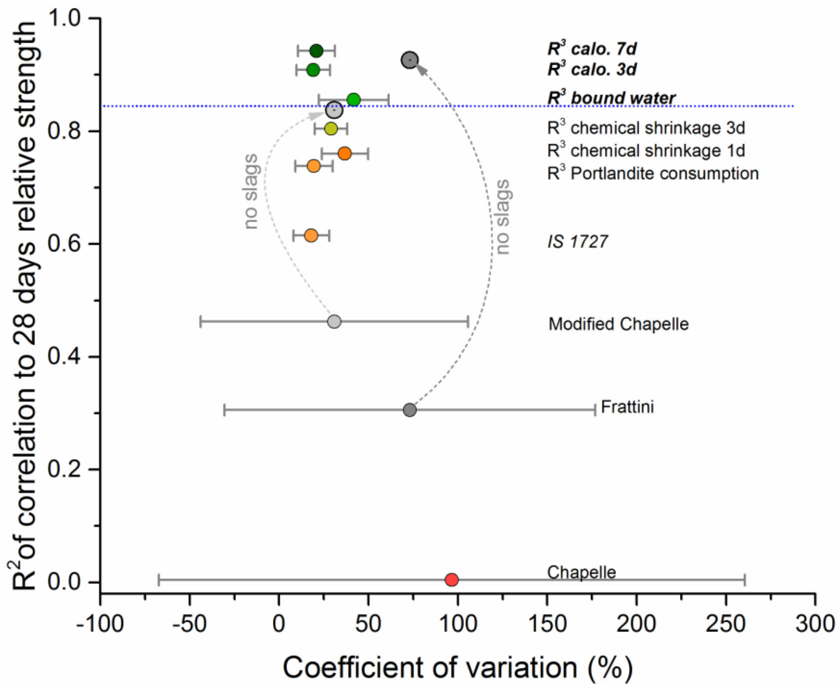

Figure 5 Correlation to 28 days relative strength vs. coefficient of variation (CV) plot, dotted blue line corresponds to R^2 value equal to 0.85, dotted grey arrows indicate the improvement of the correlation for Frattini and Modified Chapelle tests without slags.

Table 5 Summary of the methods, ranked based on the correlation to 28 days relative compressive strength.

Methods	Correlation to 28d relative strength$^{(a)}$	Coefficient of variation	Time		Equipment investment	Key equipment
			Operating	Test duration		
Units	--	%	*Hours*	*days*	relative$^{(c)}$	--
R^3 calorimetry 7 days	***0.94***	20.9	1	7	20	Calorimeter
R^3 calorimetry 3 days	***0.91***	19.1	1	3	20	Calorimeter
R^3 bound water	***0.86***	41.7	2	8	2	Oven
R^3 chemical shrinkage 3 days	0.80	29.1	4	3	2	Water bath
R^3 portlandite consumption	0.74	19.5	2	8	10	TG
IS 1727 (Indian standard)	0.62	18.1	1	10	2	Compression testing machine
Modified Chapelle	0.46	30.9	2	1	1	Reflux condenser
Frattini ([CaO] reduction)	0.31	73.1	2	8	1	Glass, pipettes
Reactive silica	0.31	-- $^{(b)}$	2	1	2	Glass, oven
Chapelle test	0.00	96.6	2	1	1	Reflux condenser

Notes: (a), R^2 of the linear fitting; (b) no data as there is only one input; (c) relative cost.

4.1 Standard reactivity test methods

Chapelle and modified Chapelle test

The Chapelle test showed no correlation to the 28 days relative strength. The Modified Chapelle test showed poor correlation (R^2 = 0.46) because that the results for slag fell out of the linear trend. When leaving out the slags the R^2 correlation coefficient is improved from 0.46 to 0.84 for the modified Chapelle test (see Figure 2 (b), Figure 5, Table 4 and Supplementary Material). The Chapelle test showed the worst reproducibility (mean CV = 96%) of all tests. The improved protocols of the modified Chapelle test resulted in significantly less dispersion of results with a mean CV of 31%. However, the committee noted that the experimental set-up is rather complex and much care is required to control the experiment and avoid carbonation.

Frattini test

The Frattini test also showed poor correlation to the 28 days relative strength. The results of slags fell out of the trend for the Frattini test (see Figure 2 (c)). When leaving out the slags the R^2 correlation coefficient is much improved from 0.31 to 0.93 (see Figure 5, Table 4 and Supplementary Material). This indicates that the Frattini method does perform well for purely pozzolanic materials, but cannot cover SCMs that show a (latent) hydraulic nature. On the other hand the Frattini test results showed a rather high CV (mean CV = 73%), which reflects the use of different local Portland cements with different alkali content.

Indian lime reactivity test (IS 1727)

The Indian standard lime reactivity test (IS 1727) showed only moderate correlation to the 28 days relative strength benchmark, but better than any other standard method when all SCMs are taken into account. For 90 days strength however the IS 1727 test performed best in terms of correlation (see Figure 2 (f) and Table 4). This may be related to the higher curing temperature of 50 °C and the longer test duration of 10 days as also for the R^3 test methods an increase in correlation was found for increased test durations (e.g. compare the R^2 for 3 and 7 days R^3 test heat release). The CV for IS 1727 is relatively good but less representative because only two laboratories used this technique at this stage, more testing is required to better constrain the reproducibility of the test.

Reactive silica test

Reactive silica test did not give acceptable correlation to the compressive strength results. The reproducibility could not be assessed as the test was only carried out by one participant.

4.2 R^3 model tests

Both the R^3 bound water and calorimetry tests gave good correlations passing the acceptance criterion. For methods compared to 28 days relative strength with R^2 higher than 0.85 (R^3 calorimetry at 7 d and 3 d, and bound water test), the linear fitting to the 28 days relative strength is shown in the plots in the Supplementary Material.

With respect early strength (7 days relative strength), the R^3 model systems perform better than the standard methods. The correlation coefficients were greater than 0.85 for all measurement methods, with the exception of the 14 days chemical shrinkage measurements. The CV for the R^3 model tests were relatively low, better than those of the standard tests.

R^3 calorimetry test

The R^3 calorimetry test showed the best correlation to 28 days relative strength with an R^2 of 0.94 for the heat release results taken at 7 days (as shown in Table 5), also the correlation to the 3 days cumulative heat was acceptable. The cumulative heat at shorter ages (0.5 and 1 day) gave the best correlation to the 7 days strength measurements. This indicates that different time intervals in the continuous measurements may be selected for correlation to the compressive strength at different ages. It can be observed in Table 4 that the R^3 heat release correlates better with the 90 days strength as the total heat is calculated at longer times. The relatively low CV indicates good reproducibility of the results. As a drawback, the equipment cost of an isothermal conduction calorimeter is relatively high. This is partially mitigated by the relatively low staff effort required compared to more laborious standard tests (see Table 5).

R^3 bound water test

The R^3 bound water test showed acceptable correlations to the 28 days relative strength with an R^2 of 0.86 for the linear correlation. Even though the linear correlation is not as good as that for the calorimetry at 7 days, the simplicity and the relatively low cost of the equipment needed (see Table 5) would enable widespread use of this test. Between the different methods for the R^3 system, the bound water test has the highest CV (42%). While the equipment used in this test is inexpensive and widely available in basic cement laboratories, the measurement protocol requires more staff effort (see Table 5). However, the technique is straightforward and does not require advanced training.

R^3 portlandite consumption test

The R^3 portlandite consumption test showed a rather weak correlation to the strength benchmark as the results of slags biased the linear trend (see Figure 3 (b)). Similarly to the Frattini and modified Chappelle tests, the correlation is much improved when the results for the slags are removed from the analysis. The relatively low CV indicates good reproducibility of the results. The current protocol requires thermogravimetric equipment which is costly, in addition the need for hydration stoppage (here by solvent exchange) makes the test rather laborious and introduces an additional source of variation.

R^3 chemical shrinkage test

The R^3 chemical shrinkage test did not give acceptable correlations to the 28 days relative strength (see Figure 3 (d)). The relationships between early age chemical shrinkage and strength appear to be non-linear, moreover later age chemical shrinkage results did not show an improvement of the correlation to the 28 days relative strength. The rather low CV indicates fair reproducibility of the results. The chemical shrinkage measurement apparatus is inexpensive, however correct execution of the measurement requires experience. Notably the loading of the containers with the paste is difficult and may strongly affect the results.

Only tests based on the R^3 system gave good performance across the whole range of SCMs investigated. Standardized methods conceived for pozzolans perform poorly when slag is included (Frattini, modified Chapelle test). Some standardized methods, e.g. reactive silica, did not show correlation to the benchmark strength development.

5 Conclusions and perspectives

This paper reports on an extensive multi-laboratory evaluation of SCM reactivity test methods carried out as part of the work of RILEM TC-267 TRM.

When taking all SCMs into consideration, all standardized methods showed poor correlation to the benchmark of 28 days relative strength. In contrast, the R^3 model calorimetry and bound water tests were able to give acceptable correlations, i.e. $R^2 > 0.85$. When slags are excluded, the correlation of the Frattini test results becomes acceptable as well. The IS 1727 test is the only method that gave reasonable correlation to the later age 90 days relative strength, possibly because of its longer duration than most other tested methods.

The Chapelle showed the worst interlaboratory reproducibility while Frattini test and modified Chapelle test had better reproducibility. The reproducibility of the R^3 model tests was the best of all the methods investigated, and can probably be improved by specifying in more detail some critical aspects in the execution of the tests.

In the phase 2 work of RILEM TC 267-TRM, the R^3 model bound water and calorimetry will be further studied due to their very promising correlations to the relative 28 days compressive strength. Further work will focus on improving the reproducibility of these methods by optimizing the test protocols. Possibilities to reduce the duration and improve correlations with early age strength development for the IS1727 test will also be included in the work for phase 2.

6 Acknowledgements

Francois Avet (EPFL, Switzerland), Luigi Brunetti (Empa, Switzerland), Nele De Belie (Gent University, Belgium), Paweł T. Durdziński (HeidelbergCement Technology, Germany), Alexandre Ouzia (EPFL, Switzerland), Olga Perebatova (Univeristy of Toronto, Canada), Yury Villagrán (Gent University, Belgium) and Bing Wang (Sinoma, China) are gratefully acknowledged for their contribution to this work.

7 Compliance with ethical standards

A. M. Joseph acknowledges the financial support from the foundations SIM (Strategic Initiative Materials in Flanders) and VLAIO (Flanders Innovation & Entrepreneurship).

S. Kramar acknowledges the financial support from the Slovenian Research Agency (research core funding No. P2–0273).

T. Sui acknowledges the support by National Key R&D Program of China (2016YFE0206100 and 2017YFB0310905) financed by the Ministry of Science and Technology of the People’s Republic of China (MOST).

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