

TECHNOTE



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Reactivity of Supplementary Cementitious Materials

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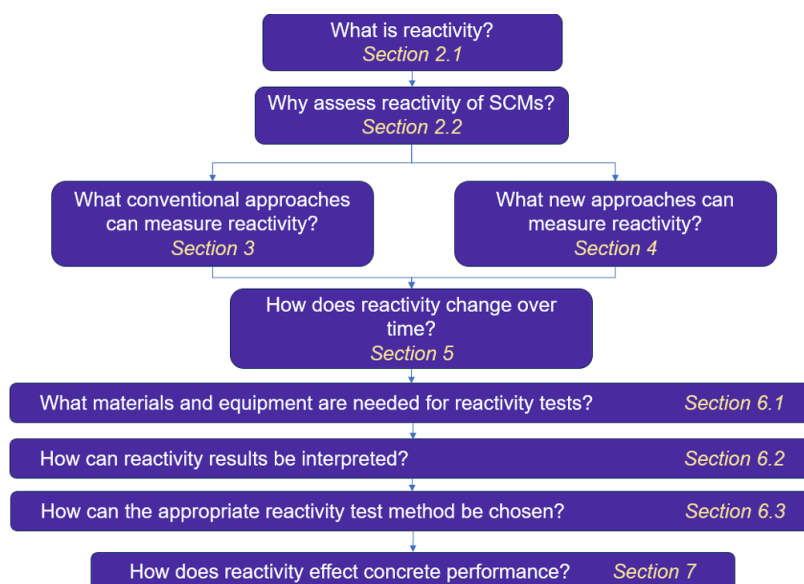
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1. MOTIVATIONS AND SUMMARY

A variety of different test methods can be used to measure or indicate reactivity of supplementary cementitious materials (SCMs). Emphasis has been placed on developing and using novel and emerging SCMs in concrete to replace cement. However, testing is needed to understand how each novel material reacts with the other constituents in a concrete mixture. Reactivity testing provides understanding about whether a material is pozzolanic, hydraulic, or not reactive enough to be considered an SCM.

The purpose of this TechNote is to support materials suppliers, producers, and infrastructure owners during materials acceptance and quality control efforts with deciding which reactivity test can be performed to characterize an SCM. In doing so, this TechNote offers background on reactivity and reactivity testing and synthesizes reactivity test methods that can be used to characterize novel materials that replace cement within concrete. Advantages, disadvantages, and challenges are discussed for the most commonly mentioned approaches for measuring reactivity. Figure 1 presents an illustration of the information provided for understanding reactivity testing of SCMs.

Figure 1. Diagram. Graphical abstract illustrating the structure of this TechNote.



Source: FHWA.

2. BACKGROUND

2.1 What Is Reactivity?

The hydration reactions between portland cement and water have been studied for more than a century. Well-known tools and standardized test methods capture the rate of reaction, degree of reaction, and their impacts on properties such as setting time and compressive strength development. SCMs, by definition,¹ participate in chemical reactions when combined with portland cement and water, but because SCMs are more variable in nature and have more complex chemistry than portland cement, the reactions are variable, and the tools and test methods for measuring the reactions are not nearly as well defined. While the reactions of portland cement are described as hydration reactions² and while cement is categorized as being hydraulic,³ SCMs' reactions are categorized as either latent hydraulic or pozzolanic.⁴ The “latent” modifier of “hydraulic,” when used to describe SCMs, refers to the reaction taking place only in the presence of a calcium-rich or alkaline solution, such as is present when cement and water are combined with the SCM.

The concept of SCM reactivity refers to how reactive the material is, independent of the type of reaction that is occurring: pozzolanic or latent hydraulic. The term “reactivity” can refer to the relative reactivity of one SCM compared with others or how much of the SCM can react (i.e., its total potential for reaction or total degree of reaction). Relative reactivity is generally captured by a measurement output such as the heat released during reaction or the amount of calcium hydroxide consumed. “Degree of reaction” refers to what percentage of the SCM can react in an alkaline solution—with the remainder considered inert. Most

common reactivity tests do not quantify the degree of reaction, but they provide measurement outputs—such as heat release or calcium hydroxide consumption—that can be used as relative measures of reactivity. Such outputs are known to correlate with concrete properties, including compressive strength. The rate of reaction—that is, reaction kinetics—is not considered in the current, common use of the term “reactivity.”

In contrast to SCMs, materials such as limestone (LS) and quarry fines are considered mineral fillers, which are expected to be unreactive or inert.⁵ However, some fillers do participate in chemical reactions. Specifically, LS is known to react with aluminate⁶ reaction products from cement hydration to form carboaluminate phases.⁷⁽³⁾ However, the extent of those reactions is significantly less than with typical SCMs, leading to LSs being generally classified as inert.

2.2 Why Test Reactivity? For What Materials Do We Measure Reactivity?

SCMs are needed for sustainable and durable concrete. SCMs were originally introduced for concrete as a method for reducing waste from other industries and mitigating alkali–silica reaction (ASR). SCMs make concrete more durable through long-term pozzolanic or latent hydraulic reactions, which refine concrete pore structure. All SCMs participate in chemical reactions when combined with portland cement. Reactivity tests can be used to verify that a material meets the definition of an SCM and provides the expected benefits to concrete. Inert fillers are not SCMs, and reactivity tests can help accurately classify materials as either fillers or SCMs.⁽⁴⁾ SCM reactivity tests typically do not measure the LS reaction to form carboaluminate phases.

¹Supplementary cementitious material: “Inorganic material such as fly ash, silica fume, metakaolin, or slag cement that reacts pozzolanically or hydraulically.”⁽¹⁾

²Hydration: “The chemical reaction between hydraulic cement and water forming new compounds most of which have strength-producing properties.”⁽¹⁾

³Hydraulic cement: “A binding material that sets and hardens by chemical reaction with water and is capable of doing so underwater. For example, portland cement and slag cement are hydraulic cements.”⁽¹⁾

⁴Pozzolanic reaction: “Reaction between calcium hydroxide and the oxides in a pozzolan, resulting in reaction products having cementitious properties similar to the products that result from the hydration of portland cement.”⁽¹⁾

⁵Mineral filler: “A finely divided mineral product at least 65 percent of which passes the 75 μm (No. 200) sieve.”⁽¹⁾

⁶Cement contains aluminate phases C_3A ($3\text{CaO} \cdot \text{Al}_2\text{O}_3$) and C_4AF ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$), which react with water and calcium sulfates to form reaction products of ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) and monosulfate ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SO}_3 \cdot 12\text{H}_2\text{O}$), also known as calcium monosulfoaluminate or AFm.⁽²⁾

⁷Hemicarbonate and monocarbonate form when monosulfate reacts with calcium carbonate in solution. The chemical formula for hemicarbonate is $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \frac{1}{2}\text{CaCO}_3 \cdot \frac{1}{2}\text{Ca}(\text{OH})_2 \cdot 11.5\text{H}_2\text{O}$, and the chemical formula for monocarbonate is $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCO}_3 \cdot 11\text{H}_2\text{O}$.⁽³⁾

Therefore, in all the tests described in this TechNote, LS is typically classified as an inert filler.

Reactivity can be considered a relative concept that classifies SCMs on scales of less reactive to more reactive. Measuring reactivity provides a qualitative indication of how these SCMs will perform in concrete mixtures. Numerous quantitative correlations between measures of reactivity, strength, and durability parameters have been shown in literature.^(5–7) The correlations occur because SCM reactions during the long term (months to years) result in calcium hydroxide consumption and pore refinement, which drive improvements in strength and most durability properties, such as ASR and sulfate attack. However, concrete performance is much more complex and depends on various factors (such as free alkali availability for ASR) when compared with reactivity, so researchers must exercise caution when extrapolating reactivity results toward durability.⁽⁶⁾

A major concern in the past decade has been the increasingly limited availability of conventional SCMs—specifically, fly ash and slag cement (called simply “slag” in the rest of this TechNote)—and thus, new SCM sources are being introduced to the concrete market. Typical SCMs are composed largely of calcia (CaO), alumina (Al₂O₃), and silica (SiO₂), with minor amounts of magnesium (MgO), iron (Fe₂O₃), sodium (Na₂O), and potassium (K₂O) oxides. The available spectrum of viable materials that could be used in concrete is vast. Given the rapid emergence of novel SCMs,⁽⁸⁾ the measurement and benchmarking of novel SCMs’ reactivity against conventional SCMs are critical. Measurement and benchmarking enable the identification of reactive materials that would likely contribute to long-term concrete durability and differentiate them from inert fillers that would not contribute to long-term concrete durability at typically used replacement levels. Measurement of reactivity enables infrastructure owners as well as the industry to rapidly identify and screen which materials should be prioritized for further testing and, ultimately, specification and use. Materials that show limited reactivity, comprehensive research, and technology transfer as SCMs might not warrant further testing and may be ready for use in the field.

As availability of conventional SCMs becomes limited, processed SCMs are becoming more common.⁽⁸⁾ Several cement and concrete companies and startups have begun large-scale manufacturing of processed SCMs, with precursors commonly being SCMs that do not meet specifications or are inert fillers. No precise definition of processing exists, and the methods used could be conventional and well-understood technologies (drying, grinding, air classification) or more complex

technologies (calcination, melting, carbon dioxide exposure). Generally, processing is performed to remove deleterious elements or phases, reduce moisture, reduce loss on ignition, and increase reactivity. If processing is performed on low-reactivity or inert materials to create SCMs, reactivity testing is a rapid way of evaluating whether such processing has indeed resulted in a reactive SCM.

Reactivity testing is important and helpful with regard to benchmarking materials. The performance of SCMs, such as fly ash and slag, as well as inert fillers, such as LS, in concrete is well known. Generally, if novel SCMs have chemistry and reactivity similar to known, conventional SCMs, the novel SCMs’ performance in concrete is expected to be similar to that of the corresponding conventional SCM. Reactivity is an important measure for guiding the use of SCMs in concrete because reactivity affects mechanical property development, pore refinement, and aspects of chemical durability performance, and no other measure can capture all of those.

Reactivity should be measured when new materials are classified for use and on a regular basis during materials’ production and distribution. Measurement of reactivity on SCMs that have longstanding use in concrete mixtures, such as fly ashes, is also worthwhile since their inherent plant-to-plant and batch-to-batch variability affects reactivity and, thus, performance. The combining of reactivity with compositional and mineralogical data yields a good understanding of how SCMs affect concrete.

3. CONVENTIONAL APPROACHES TO MEASURE REACTIVITY

3.1 Overview of Relevant ASTM Specifications

Currently, SCMs are specified under several ASTM International standards, including ASTM C618 for coal ash and natural pozzolans,⁽⁹⁾ ASTM C989 for slag,⁽¹⁰⁾ ASTM C1240 for silica fume (SF),⁽¹¹⁾ ASTM C1866 for ground glass,⁽¹²⁾ and ASTM C1697 for blended SCMs.⁽¹³⁾ ASTM C1709 is a guide for evaluating alternative SCMs irrespective of origin.⁽¹⁴⁾ None of these documents currently specify direct reactivity testing of SCMs—meaning, the measurement of reactants consumed or reaction products formed. Instead, the ASTM International standards rely on indirect evaluations of reactivity.

An example of an indirect means of assessing reactivity is the control of composition. ASTM C618 specifies that the sum of the primary oxides (SiO₂ + Al₂O₃ + Fe₂O₃) must exceed 50 percent for coal ash and 70 percent for natural

pozzolans.⁽⁹⁾ ASTM C1240 specifies that the SiO₂ content must be greater than 85 percent.⁽¹¹⁾ Those requirements are indirect reactivity assessments because silica and alumina participate in pozzolanic reactions. Therefore, having a high content of those oxides suggests that the material may be reactive. However, given that finely ground silica sand (quartz, also called silica flour) has a sum of oxides greater than 95 percent but is not a reactive SCM at ambient construction temperatures, the sum of oxides alone is inadequate for assessment of the reactivity of an SCM. A compositional requirement for reactivity cannot only specify oxides but also must address the phase composition, the measurement of which is beyond the scope of current ASTM International standards.

ASTM International standards also control the fineness of coal ash, natural pozzolans, slag, SF, and ground glass by limiting the amount retained on the 45-mm (No. 325) sieve under wet sieving. (See references 9–12.) Materials with higher finenesses are likely to be more reactive, so fineness is an indirect measure of reactivity. However, fineness is not sufficient on its own to assess reactivity because composition plays a dominant role in reactivity.^(7,8)

3.2 Strength Activity Index

The strength activity index (SAI) in ASTM C618 has long been considered a reactivity test because, generally, more reactive SCMs lead to higher compressive strengths for mortars made with the SCM.^(9,15) However, mortar strength is dependent on factors beyond SCM reactivity, so the SAI test is an indirect reactivity assessment, not a direct reactivity measurement. The SAI test has many shortcomings that have been discussed by several researchers. (See references 4 and 16–18.) Perhaps the biggest shortcomings of the SAI for assessing SCM reactivity are the age of testing (7 or 28 d), relative strength threshold (75 percent of the control when using a 20-percent replacement of cement by SCM), and the fact that testing is performed on mortars at variable water to cementitious materials ratios (w/cm) to achieve constant mortar flow. Many pozzolanic SCMs, including Class F fly ash (FFA), are slow reacting by nature, and their impacts on strength may not be apparent under room temperature curing until 56 d, if not longer. Also, a 75-percent relative strength threshold is too low to assess the impact of a 20-percent SCM replacement since dilution alone (with quartz, for example) can result in relative strengths greater than 80 percent.⁽⁴⁾ The ASTM C1797 specification, which is applicable for ground LS and aggregate mineral fillers, uses the same 75-percent limit at 20-percent replacement.⁽¹⁹⁾ The variable w/cm is problematic in assessing the impact of SCMs on strength because the w/cm has a significant impact on

strength independent of SCM reactivity. The variable w/cm (constant mortar flow) in the standard was intended to couple assessment of water demand with impact on strength, but ultimately, increases challenges in the use of SAI to assess reactivity. Clearly, both water demand and strength are important parameters to measure, but with recent technological advances, including superplasticizers, the coupling of the two measures in ASTM International specifications must be revisited.

3.3 Slag Activity Index and Accelerated Pozzolanic SAI

ASTM C989 contains a slag activity index using the compressive strength of a mortar containing 50-percent slag and 50-percent cement by mass.⁽¹⁰⁾ The slag activity index is used to classify slags into grades 80, 100, or 120, depending on the resulting relative strength at 28 d. The assumption is that greater reactivity results in higher strength. This reactivity assessment, while indirect, achieves its purpose for this class of materials since slags have more limited variability—compared with coal ashes and natural pozzolans—in factors that affect reactivity and strength such as composition, shape, size, and porosity. The same is true for ASTM C1240, which contains an accelerated pozzolanic SAI test involving compressive strength testing of a mortar with 10-percent SF replacement of cement by mass.⁽¹¹⁾ The threshold of 105-percent compressive strength at 7 d for mortars with SF compared with those without is likely a sufficient indirect indicator of reactivity, given the limited number of variables in SF that would affect strength beyond its pozzolanic reactivity.

3.4 European EN 197 Test Method

Most existing standardized reactivity tests for SCMs assess the performance of the SCM in a system that is intended to mimic, at least to a certain extent, a portland cement pore solution or paste. One exception is the European Standard 197-1, which defines the reactive silica content of an SCM by using a series of acid and base treatments.⁽²⁰⁾ The method uses composition determined by dissolution-based experiments to indirectly infer reactivity. The test has been criticized for the testing conditions that are not representative of pore solutions and, therefore, do not assess the solubility of silica and alumina.⁽²¹⁾

3.5 Chapelle and Modified Chapelle Tests

The Chapelle test is a rapid, direct measure of the pozzolanic reaction that measures CaO (calcium oxide or lime) consumption by a pozzolan in solution at an elevated temperature. The test was originally developed in 1958,⁽²²⁾ and a standardized version is published in the French standard NFP 18-513 (2010).⁽²³⁾ In the French

standard, 1 g metakaolin is combined with 2 g lime (prepared through the calcination of calcium carbonate (CaCO_3)) in 250 mL distilled water and kept at $85 \pm 5^\circ\text{C}$ in an Erlenmeyer flask, constantly stirred, on a heating plate for 16 h. Evaporation is prevented by attaching a reflux condenser. After cooling and filtering, the remaining calcium in solution is determined by titration with hydrochloric acid by using a phenolphthalein indicator.

The Chapelle test has the advantage over many other reactivity tests in that it provides conclusive results in less than 24 h. However, the test is useful only for pozzolanic materials like siliceous fly ashes, calcined clays (CCs), SF, and ground glass because such materials react with calcium hydroxide to form calcium silicate hydrate (C-S-H) and are not relevant for latent hydraulic SCMs. The test is sensitive to particle size for some metakaolins⁽²⁴⁾ and for pumice (P),⁽²⁵⁾ showing higher values for smaller particles. The results correlate well with other indexes for pozzolanicity such as compressive strength and calcium hydroxide consumption,^(25,26) both of which take a considerably longer time to measure. Certain important concerns have arisen regarding Chapelle test results. Quartz, which is not pozzolanic, results in measurable lime consumption, which leads to an erroneous suggestion of pozzolanicity.^(21,25) The suggestion is due to the high temperature used in the test ($85 \pm 5^\circ\text{C}$), which enhances dissolution of the crystalline silica. Calcium-bearing, latent hydraulic SCMs, such as slag or Class C fly ash (CFA), will not produce correct results in tests that examine calcium hydroxide consumption since the SCM itself contributes calcium to the solution.⁽²¹⁾ Additionally, latent hydraulic SCMs do not need calcium hydroxide for their reaction; thus, although their calcium hydroxide consumption might be low, their performance in concrete might be better than is suggested by the Chapelle test. While intralaboratory repeatability can be quite good (approximately 5-percent measurement error for replicate samples),⁽²⁵⁾ the interlaboratory repeatability of the test has been questioned (mean coefficient of variation of 31 percent)⁽²⁶⁾—perhaps due to the complexity of the test setup and the risk of carbonation during testing.^(21,27)

3.6 Frattini Test

In the Frattini test, in the European Standard EN 196-1 Part 5, a suspension containing the SCM, portland cement, and calcium hydroxide is cured for at least 7 d at 40°C .⁽²⁰⁾ After cooling to room temperature, the suspensions are filtered, and titration is used to measure calcium and hydroxyl ion concentrations. Solutions with calcium concentrations below saturation level suggest that the SCM has consumed calcium hydroxide. Similarly to the Chapelle test, the complicated process can lead to poor interlaboratory reproducibility, and the test is not

appropriate for calcium-bearing SCMs. In addition, the test is sensitive to the cement used since cements have different reactivities themselves and different calcium hydroxide amounts produced through hydration.^(22,27)

3.7 Lime Reactivity Test

Lime reactivity tests have long been used to assess SCM reactivity and are currently standardized in Canada (CSA A3004-E1) and India (IS 1727-1967).^(28,29) A version of a lime reactivity test was previously included in ASTM C311-87a (1987)⁽³⁰⁾ but was withdrawn and does not appear in the current ASTM C311 standard.⁽¹⁵⁾ Several studies recently examined the effectiveness of the Indian standard lime reactivity test for the test's promise in evaluating SCM reactivity.^(26,27) In this test method, SCMs are mixed with calcium hydroxide and Indian standard sand in fixed ratios. (See references 26–29.) The water content is adjusted to achieve a specific mortar flow. Cubes are cured and sealed at 27°C for 48 h and 50°C at 90 ± 5 percent relative humidity for an additional 6 d before testing in compression. One of the challenges with this test involves the variable water content needed to achieve specific mortar flow,⁽¹⁴⁾ which makes interpreting the results a challenge. A major advantage of this test is that many labs are equipped to measure strength, so no unusual equipment is needed. In addition, identifying inert materials is straightforward because cubes with inert materials would not harden or have any measurable strength in the test. In principle, inert materials could even be identified visually based on lack of hardening. The test is appropriate for both pozzolanic and latent hydraulic materials since both will result in measurable strength.

4. NEW APPROACHES TO MEASURE REACTIVITY

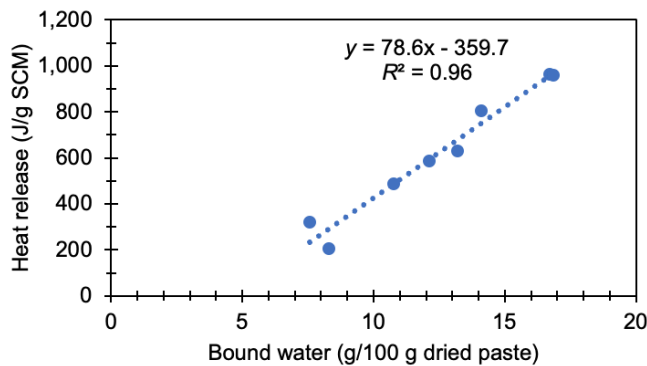
4.1 Standardized Method: ASTM C1897 (R^3 Test)

ASTM C1897-20 is a recently standardized SCM reactivity test that is increasingly used by the research community and the industry.⁽³¹⁾ It is based on the rapid, relevant, and reliable (R^3) test.⁽⁵⁾ The R^3 test was originally developed as a means of predicting the compressive strength of cement mortars containing calcined kaolinitic clays. RILEM Technical Committee (TC) “267-TRM: Tests for Reactivity of Supplementary Cementitious Materials (SCMs)” then formed to evaluate the applicability of the R^3 test—compared with other standardized reactivity test methods—for assessing reactivity for a broader range of SCMs.⁽²⁷⁾ Participants in the TC also refined the test parameters, evaluated variables and robustness (e.g., mixing, temperature, and mixture composition),⁽³²⁾ and developed thresholds to

distinguish inert fillers from SCMs by comparing R^3 results with compressive strength.⁽³³⁾

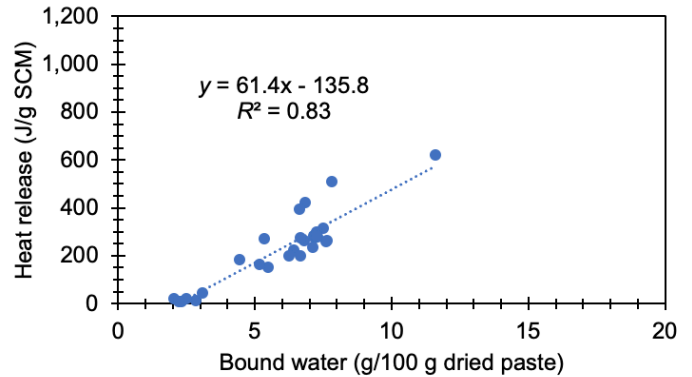
In the R^3 test standardized in ASTM C1897-20, a paste is made containing the SCM, calcium hydroxide ($\text{Ca}(\text{OH})_2$), CaCO_3 , and a potassium sulfate (K_2SO_4) solution in fixed proportions.⁽³¹⁾ ASTM C1897 provides two methods for assessing reactivity using the R^3 paste. In method A, the sample is placed in an isothermal calorimeter for 7 d at 40 °C, and the heat release is recorded. In method B, the sample is sealed and cured at 40 °C for 7 d, dried, ground, and heated at a controlled rate from 40 °C to 350 °C, and the mass loss during heating is recorded as the bound water. Both methods produce highly repeatable results. Researchers generally prefer method A because less labor is involved and continuous data are obtained. The industry generally prefers method B because the capital cost of equipment is lower and the equipment involved is less complex. A strong correlation exists between the method A and method B results—particularly for a given class of SCMs, as shown in figure 2 for CCs ($R^2 = 0.96$). The correlation between methods A and B is less strong when a variety of SCMs are tested (figure 3) but still sufficient to allow interchangeable use. The reduction in correlation is because reactions in SCMs of different compositions have different calcium aluminosilicate contents, resulting in differing enthalpies and reaction products with variable bound water contents.

Figure 2. Graph. Comparison of method A (heat release) and method B (bound water) values from ASTM C1897 (the R^3 test) for calcined kaolinitic and smectic clays.⁸⁽³¹⁾



Source: FHWA.

Figure 3. Graph. Comparison of method A (cumulative heat release) and method B (bound water) values from ASTM C1897 for a variety of inert materials and reactive SCMs (adapted from reference 33).⁽³¹⁾



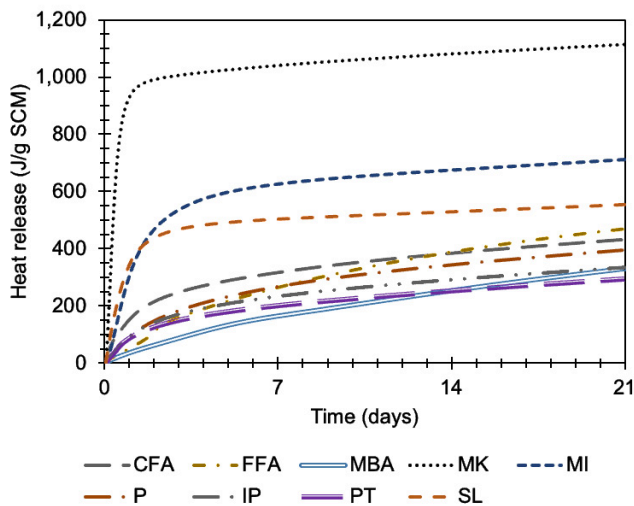
Source: FHWA.

The R^3 test is increasingly accepted as a reliable means to characterize the reactivity of SCMs. The R^3 test is a reactivity test, not a pozzolanicity test because it does not distinguish between pozzolanic reactions, latent hydraulic reactions, or other reactions.⁽³⁴⁾ The reactivity of slow-reacting SCMs such as coal ashes and some natural pozzolans may be underestimated in this test method because they continue to show substantial reaction after the 7-d testing period, as shown in figure 4.⁽³⁴⁾ However, a correction for kinetics could be made, for example, by using the ratio of the 3-d to 7-d heat release; or heat release data could be extrapolated using the data from the last day for slowly reacting materials.⁹ Extending the test for longer than 7 d could provide useful results, as shown in figure 4, but given that heat flow values tend to reduce to values close to those of the noise, concern has arisen regarding data quality when testing past 7 d. Given that heat release is challenging to extend past 7 d, the bound water presents an advantage, whereby multiple measurements of bound water (1, 3, 7, 21 d) might present a clearer picture of reactivity. When performance in other measurements is promising or when the 3-d to 7-d heat release (or bound water) ratio is low, producers may consider testing reactivity past 7 d.

⁸Previously unpublished data from K. O'Quinn, graduate research associate, University of Texas at Austin.

⁹As an example, in figure 4, FFA has a 7-d heat release of 264 J/g SCM and a 6-d heat release of 241 J/g SCM, which gives an average heat release of 23 J/g SCM/d. On the other hand, the faster reacting metakaolin (MK) has values of 1,040 J/g SCM and 1,034 J/g SCM at 7 d and 6 d, respectively, with the corresponding average heat release being 6 J/g SCM/d. The heat release at 21 d can be estimated by assuming the same constant heat release over 7 to 21 d. Using this method suggests values of 21-d heat release of 586 J/g SCM for FFA and 1,124 J/g SCM for MK. The actual values are 469 J/g SCM and 1,114 J/g SCM, respectively, thereby showing the tendency of this extrapolation method to provide an indication of later age reactivity but to overestimate reactivity results. The discrepancy between the estimated and actual values for FFA suggests that a more sophisticated method for assessing the kinetic effect is needed.

Figure 4. Graph. Extension of calorimetry testing (method A) in ASTM C1897 beyond 7 d (adapted from reference 33).



Source: FHWA.

IP = impure pumice; MBA = milled bottom ash; MK = metakaolin; MI = impure calcined kaolinitic clay; PT = pumiceous tuff; SL = slag.

An additional concern with the test is that the sulfates added to the system in the R^3 paste can lead to oversulfation, in turn leading to modification of the reactivity for some SCMs.⁽³⁴⁾

4.2 Modified R^3 method

The R^3 test is performed at 40 °C and uses a rather complex pore solution ($\text{Ca}(\text{OH})_2$, KOH [potassium hydroxide], K_2SO_4 , and CaCO_3).⁽³¹⁾ Modified variants of the R^3 test have been proposed. One modified version, known as the “modified R^3 test” or the “pozzolanic reactivity test (PRT)” uses a mixture of SCM and calcium hydroxide in a 1:3 mass ratio with a 0.9 liquid/solid ratio. (See references 35–38.) The liquid used is a 0.5 M KOH solution as a simplified pore solution. After mixing, heat release is obtained for 10 d in an isothermal calorimeter at 50 °C, and at the end of 10 d, thermogravimetric analysis (TGA) is performed to measure calcium hydroxide consumption; bound water can also be measured. By plotting heat release versus calcium hydroxide consumption, materials can be classified. Example results are shown in figure 5, with general classifications of inert, pozzolanic, highly pozzolanic, and hydraulic. The LS and basalt fines (BF) are examples of inert materials. For such materials, the heat release and calcium hydroxide consumption are very low (typically below 100 J/g SCM and 30 g/100 g SCM, respectively). On the other extreme are SF and CC, which are highly pozzolanic: typically, heat release >500 J/g SCM and calcium hydroxide consumption

>100 g/100 g SCM. CC reactivity depends heavily on clay composition and processing, with heat release increasing linearly as the kaolinite content increases up to approximately 1,000 J/g SCM.⁽³⁹⁾ The heat release values of the pozzolanic CFA, Class F fly ash (FFA), and P are similar—approximately 300 J/g SCM—but the calcium hydroxide consumption varies from 40 to 100 g/100 g SCM; the more siliceous materials have higher calcium hydroxide consumption. For other materials tested, a larger range of heat release—100–400 J/g SCM—has been seen. As discussed later, these materials also have different reaction kinetics. The slag has heat release of approximately 450 J/g SCM and calcium hydroxide consumption of approximately 35 g/100 g SCM and shows hydraulic behavior due to high calcium content. The heat release is strongly influenced by SCM fineness, chemical composition, and mineralogy.^(35,36,40)

The outputs of this test (heat release and calcium hydroxide consumption) can be used to quantify the maximum degree of reactivity (DOR*) for a given SCM. That DOR* is determined by comparing the measured outputs with theoretical values of heat release and calcium hydroxide consumption for reactions of pure SiO_2 and Al_2O_3 and interpolating to compute DOR*.^(37,38) The DOR* is useful as an input for thermodynamic modeling, which can help predict concrete properties.

Differences between the R^3 test and the modified R^3 test have been explored.⁽⁴⁰⁾ Importantly, all tested fly ashes were reactive using both tests, and researchers are in broad agreement between the tests. Thus, for first approximation, either test can be used. The modified R^3 test, performed at 50 °C, typically shows a higher early-age heat release than the R^3 test, run at 40 °C. Typically, after the first 3 d, due to additional reactions occurring with the sulfates and carbonates present in the R^3 system (leading to the formation of monosulfate, hemihydrate, monocarbonate, and other phases) but not in the modified R^3 test, the heat release in the R^3 test exceeds that of the modified R^3 test. The time of this crossover was shown to inversely correlate with SCM $\text{CaO} + \text{Al}_2\text{O}_3$ content. For SCMs with high $\text{CaO} + \text{Al}_2\text{O}_3$ contents, the heat release may always be higher in the R^3 test. On the other hand, for highly siliceous SCMs such as P, the heat release may always be higher in the modified R^3 test.

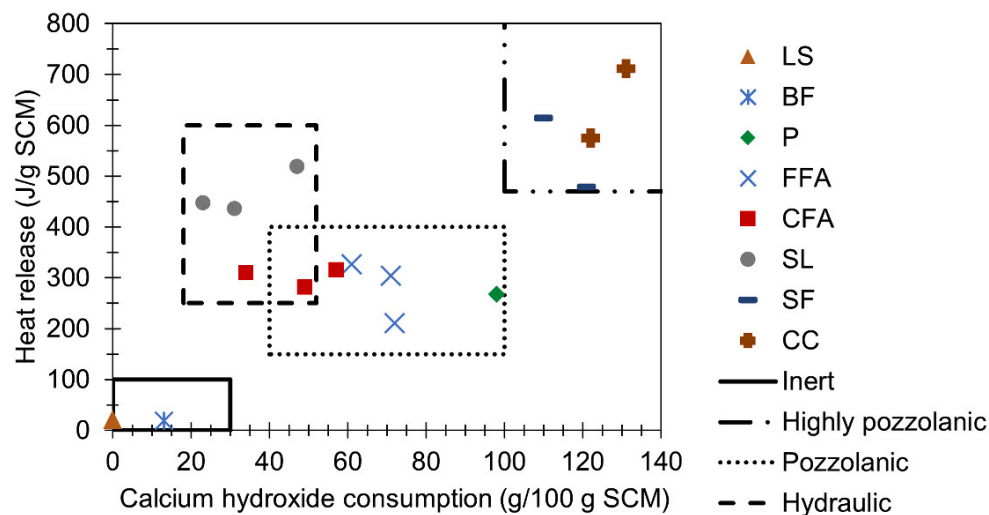
One of the advantages of the modified R^3 test is that the measurement of calcium hydroxide consumption allows for better classification. However, researchers have proposed various linear relationships between heat release and calcium hydroxide consumption. For different materials, researchers have related the slopes of those relationships to the SCM chemical

composition. Therefore, if the chemical composition is known, researchers can estimate calcium hydroxide consumption based on heat release or vice versa, which means that running two measurements is unnecessary.⁽³⁶⁾ Figure 5 makes evident that if the high CaO materials are removed, the heat release increases roughly linearly with the calcium hydroxide consumption. Alternatively, if one wanted to get information about pozzolanicity and separate out pozzolanic and latent hydraulic materials, calcium hydroxide consumption can be performed as part of the R^3 test as well. Similarly, the test duration of the R^3 test could be adjusted to be longer for slowly reacting siliceous SCMs, or kinetic corrections can be established. The only true advantage of the modified R^3 test thus appears to be the simplified pore solution. The amounts of carbonates and sulfates that participate in SCM reactions in cementitious systems are unclear, so which pore solution composition is more appropriate is also unclear. However, the modified R^3 test is easier to perform because of the simplified pore solution.

While significant data using the modified R^3 test are available, the test is not standardized. Arguably, given that the modified R^3 test is a relatively simple modification of the R^3 test, standardization of this test is not likely to be particularly advantageous. Another major disadvantage is that isothermal calorimetry

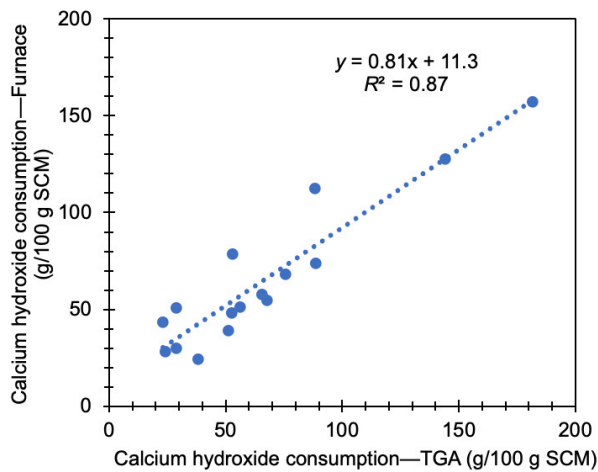
and TGA are expensive and complicated to use. Heat release from isothermal calorimetry and bound water determined from a furnace correlate, as discussed in section 4.1. More recent research has shown that calcium hydroxide consumption determined from TGA and calcium hydroxide consumption estimated from furnace mass loss measurements also correlate (figure 6).⁽⁴¹⁾ While variability increased and data quality decreased, it appears that a furnace can be used to determine both bound water and calcium hydroxide consumption in R^3 and modified R^3 systems.^(16,41) While the furnace-based test is promising, robustness and round robin data are absent. Researchers also identified that the cooling procedure after the sample is removed from the furnace has a strong impact on the measured reactivity values. The effect of cooling is likely because of the formation of reactive calcium oxide from the decomposition of calcium hydroxide, which could react to form calcium hydroxide or CaCO_3 under ambient conditions, which affects the mass measurements. Further study of cooling effects is needed.⁽⁴¹⁾ Research has already shown that heat release and bound water correlate. Correlations between heat release and calcium hydroxide have been discussed. However, using a furnace to measure calcium hydroxide consumption is a useful alternative when TGA is not available.

Figure 5. Graph. Heat release versus calcium hydroxide consumption using modified R^3 test for LS, BF, P, FFA, CFA, slag, SF, and CC (adapted from references 34, 35, 39).



Source: FHWA.

Figure 6. Graph. Calcium hydroxide consumption values determined from a furnace correlate with those determined from TGA (adapted from reference 40).



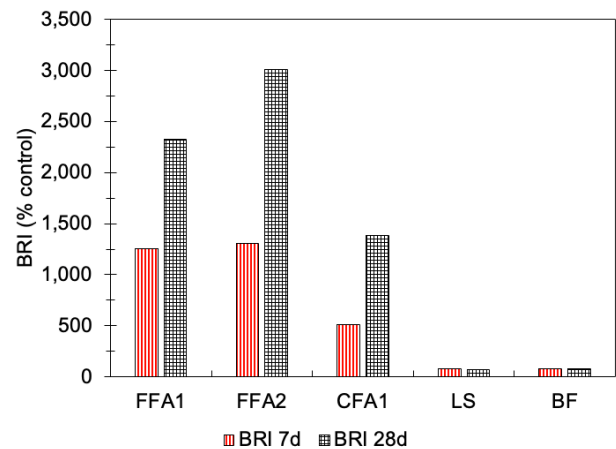
Source: FHWA.

4.3 Bulk Resistivity Index Test

Electrical resistivity of concrete is a measure of concrete's opposition to the movement of charged ions under the action of an electrical field. Electrical resistivity can be measured on the bulk material or on the surface of a sample. Electrical resistivity is affected by concrete pore volume, pore sizes, pore connectivity, pore solution composition, degree of saturation, and specimen temperature. The electrical resistivity of concrete is generally a quality and durability indicator. Higher resistivity values indicate that the concrete will generally show good long-term performance.^(42,43)

The use of SCMs generally increases bulk and surface resistivity when compared with control mixtures without SCMs, whereas inert fillers such as LS do not significantly increase resistivity. The increase is due to SCMs' refinement of pore structure and because they reduce pore solution ionic concentrations. However, for many SCMs, the increase becomes apparent only after long-term curing (56 d).⁽³⁴⁾ Because long-term curing is not a feasible option for standardization or specification, some have suggested using higher temperature curing or higher replacement. That resulted in the development of the bulk resistivity index (BRI) test, wherein the bulk resistivity of mortar mixtures at 7 and 28 d is compared with the mortar mixtures of the control.⁽⁴⁴⁾ Ideally, the curing is performed at 50 °C for a mortar with 30 percent or 50 percent SCM replacement and a constant *w/cm*. SCMs increase bulk resistivity significantly (up to 3,200 percent when compared with the resistivity of a control mixture with no SCMs), whereas inert materials do not; example results are shown in figure 7.⁽⁴⁴⁾

Figure 7. Graph. BRI (bulk resistivity expressed as a percent of control) for reactive SCMs (FFA1 and FFA2 are FFAs, CFA1 is CFA) and two inert fillers. Results are shown at 7 and 28 d for a mortar with *w/cm* = 0.485, 50 percent SCM replacement of cement, and curing temperature 50 °C (adapted from reference 44).



Source: FHWA.

Because resistivity is sensitive to reactivity, bulk resistivity at higher temperatures can differentiate inert and reactive materials.

The BRI does not differentiate pozzolanic and latent hydraulic materials, although that differentiation can be determined by dividing SCM composition by CaO content and using a threshold level of CaO for differentiation. Neither robustness testing nor round robin testing has been performed on this test. Resistivity is likely to become a common measurement for departments of transportation, making this method attractive—especially because it could be performed on the same mortar cubes used for SAI. Doing so could enable SAI to become a report-only value and should be a relatively simple change to make to the ASTM C618 specification.⁽⁹⁾ Surface resistivity is known to correlate with bulk resistivity,⁽⁴⁵⁾ and therefore, surface resistivity could also be used instead of bulk resistivity, though the specimen geometry and Wenner probe configuration of typical surface resistivity equipment would have to be accounted for. The feasibility of reducing test duration to 7 d and the use of more standardizable temperatures—such as 38 °C—are being explored.

A major advantage of this test is that it is relatively easy to perform. All that is needed is a bulk resistivity meter, which is advantageous because the meter could also be used to assess the durability of concrete toward chloride ingress. Another advantage is the simple differentiation between reactive and inert materials

due to the sensitivity of resistivity to reactivity. Major disadvantages of the BRI test are lack of robustness, lack of round robin data, and limited data available for SCMs, including high CaO latent hydraulic slags—apart from the ones studied in reference 44. The effects of varying experimental conditions such as temperature, curing (limewater versus pore solution), and SCM replacements on BRI results are not completely clear.

4.4 Compressive Strength

The shortcomings of the SAI test in ASTM C618, which follows the methods in ASTM C311,^(9,15) were discussed briefly in section 3.2. The concern with using an SAI test to determine SCM reactivity is that the test method does not accurately distinguish inert fillers from reactive SCMs.⁽⁴⁾ The equivalent test in the European Standards (EN 450-1) addresses some of the problems with SAI by fixing the w/cm at a constant value, requiring 28- and 90-d testing using 25 percent SCM by mass and specifying a relative strength threshold of 85 percent at 90 d.⁽²⁰⁾ Al-Shmaisani et al. modified the SAI test to more closely match the EN 450-1 standard by increasing cement mass replacement to 25 percent, holding w/cm constant, and testing up to 90 d.⁽³⁴⁾ The authors of that study found that an inert quartz filler still passed the test at a 25-percent cement replacement and an 85-percent relative strength threshold at 28 d. However, the quartz did not pass the test at 90 d, suggesting that any attempt to use compressive strength to assess SCM reactivity would require 90-d testing.

Some have suggested that mitigation of ASR could be used to assess SCM reactivity since pozzolans reduce expansion due to ASR. A recent study⁽⁴⁶⁾ demonstrated that accelerated mortar bar tests for ASR (ASTM C1567)⁽⁴⁷⁾ are just as prone to false identification of inert fillers as SAI tests. Interestingly, the authors found that combining the two tests can correctly predict SCM reactivity. If the cement replacement by SCM is high enough to control ASR expansion and if the replacement also results in 75 percent relative strength, it is reactive; if the SCM amount needed to control ASR does not reach 75 percent relative strength, it is inert. Neither test is sufficient on its own using 14-d expansions and 7- or 28-d strengths, but together, they may be able to provide a preliminary reactivity assessment.

4.5 Lime Strength Test

Recent interest in evaluating pozzolanicity tests is resulting in renewed use of the lime reactivity test.^(25,26) Kasaniya et al.⁽⁴⁷⁾ identified certain shortcomings of the test method in the Canadian standard method

(CSA A3004-E1).⁽²⁸⁾ Of particular concern was that the lime-to-pozzolan and water-to-binder ratios were not fixed and are determined by the specific gravity of the pozzolan and mixture flow, respectively. To address those problems, the study's authors examined several variables in the test.⁽⁴⁸⁾ Ultimately, the authors fixed a w/cm ratio of 0.65 by mass, with the addition of a high-range water reducer, to control flow. The authors fixed a sand-to-binder mass ratio of 2.5. Compressive strength testing demonstrated that the lime-to-pozzolan ratio had a much greater effect on results for metakaolin than for other SCMs (30 tested in total), with strength decreasing as the lime-to-pozzolan ratio increased. The authors then chose a ratio of 1:1.5 as optimum. The authors also chose a solution based on that used for R^3 testing—containing 0.3 M K^+ solution with K_2SO_4/KOH ratio of 5 and calcite at a 1:15 calcite-binder ratio—as optimum. With respect to curing temperature, the authors found that compared with 50 °C, a temperature of 40 °C increased strength and reactivity.

The lime reactivity test shows promise as a method to identify reactive SCMs and screen out inert fillers and can even be used as an alternative to the R^3 test method since the curing conditions and pore solution composition are similar; this alternative could be of interest to labs that do not have access to calorimeters or furnaces but have compressive-strength-testing equipment.⁽³⁴⁾ The drawbacks to the lime reactivity test are similar to those of the R^3 test in that the lime reactivity test is a general reactivity test, not a pozzolanicity test, and may underestimate the performance of slow-reacting SCMs.

4.6 Pozzolanic Activity Test

A further modified version of the Chapelle test is currently being called the “pozzolanic activity test.” The Chapelle test is different from most SCM reactivity tests in that it is a direct measure of the extent of pozzolanic reaction since it measures the consumption of calcium hydroxide in solution. As discussed earlier, the test was developed for testing metakaolin^(22,23) and is not appropriate for calcium-bearing SCMs because calcium-bearing SCMs contribute calcium to the solution and disrupt the test. Neither does the test measure hydraulic or latent hydraulic reactions in SCMs. The Natural Pozzolan Association in the United States has been working with a metakaolin producer to develop and evaluate the simplified version of the Chapelle test—the pozzolanic activity test—specifically for raw and calcined natural pozzolans. The test is currently undergoing development and round robin testing at ASTM International but is not yet standardized. The

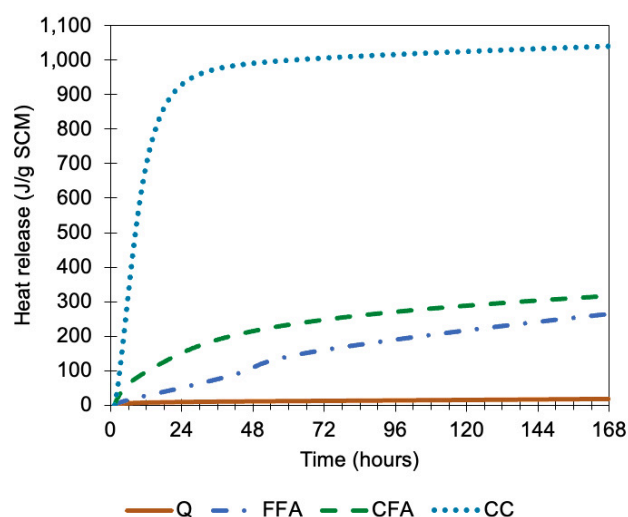
modifications aim to simplify the procedure to encourage broader use in the community and reduce interlaboratory testing errors. In this method, the SCM is combined with a calcium hydroxide solution in a screw-top plastic Erlenmeyer flask and then placed in a 70 °C oven for 48 h. The titration to determine calcium hydroxide consumed is the same as in the original Chapelle method. If the method is proven to be repeatable and reliable, the method may become standardized.

5. CONSIDERATIONS

5.1 Kinetics of Reactivity

The reactions of a cementitious system occur continuously. Therefore, the reactivity changes with the age of the cementitious system at the time of measurement. The kinetics of reactivity is the rate of change of any reactivity measure. The impact of kinetics is shown in figure 8 for the R^3 test.⁽³⁴⁾ Figure 8 shows an inert material with the heat release always low (Q = quartz), a slowly reacting material (FFA in this case), a faster reacting material (CFA in this case), and a fast-reacting and highly reactive material (CC—a high-range CC). The differences between LS and CC are evident and consistent at all times, but the differences between FFA and CFA are more complex (heat release of CC is greater than the heat release of FFA and CFA, which are greater than the heat release of LS). Specifically, FFA initially shows lower reactivity than CFA, but the reactivity of FFA is sustained, and at later ages, the heat releases by FFA and CFA are approximately equal. FFAs and other siliceous SCMs, such as P and other natural pozzolans, show slow but sustained heat release, also evident in figure 4.^(36,40) Their heat release is often lower than those of calcium aluminosilicate SCMs (CFA) at early ages but not necessarily at later ages. Therefore, the entire heat release curve must be considered because it is reflective of how these materials perform in concrete; that is, siliceous SCMs show lower early-age compressive strength than the calcium aluminosilicates, but that may not be true at later ages. The kinetics of reactivity are critical when a measure such as bound water is used. Because bound water is a single-point measure, it does not provide kinetic information and, therefore, provides an incomplete picture of SCM reactivity. If the objective is to differentiate reactive SCMs and inert fillers, then a single measurement of bound water would be adequate. To better understand the SCM reactivity, a two-point bound water measurement (measured at 1 d and 7 d as an example) can provide an indicator of kinetics. Specifically, slowly reacting materials would have a higher ratio of 7-d/1-d bound water than fast-reacting materials.

Figure 8. Graph. Heat release using the R^3 test for Q = quartz, FFA, CFA, CC = high-range CC (adapted from reference 33).



Source: FHWA.

A comparison of several materials such as CFA, slag, and metakaolin using the R^3 test or the modified R^3 test shows that many reactive materials release approximately 85 percent of their 7-d or 10-d heat release at 3 d.^(34,39) Thus, the test duration can be reduced to 3 d for these materials, with the optional use of a kinetic factor. The major exceptions are the slowly reacting siliceous SCMs, such as FFA and P, which have much lower percentages of their 7-d or 10-d heat release at 3 d. For the siliceous SCMs, such as FFA and P, a different kinetic correction should be used, or the test should be continued longer—potentially up to 14 d. For the materials shown in figure 8, the 3-d/7-d heat release values are 80 percent for the CFA and 97 percent for the CC, but only 61 percent for the FFA, suggesting that stopping the test at 3 d could be acceptable for CFA and CC but not for FFA.

5.2 Discrete Versus Continuous Testing

Because a cementitious system's reactions occur continuously, reactivity can be measured continuously through a period of time or in discrete measurements at a single point in time. For some test methods, discrete measurements may occur at multiple points in time to provide an indication of how the reactivity is changing. The isothermal-calorimetry-based R^3 and modified R^3 tests are continuous and give heat release curves during 1–10 d. In most cases, as discussed in section 5.1 and elsewhere, stopping the test by 3 d could be acceptable. On the other hand, the SAI, BRI, lime strength, Chapelle, and Frattini tests are discrete in that they provide a single value at a single point in time for characterization. Similarly, using

calcium hydroxide consumption or bound water (using a furnace or TGA) in the R^3 or modified R^3 test is also discrete. To account for reactivity kinetics, obtaining multiple-point measurements (two or three) becomes important for the discrete tests. Performing each test multiple times to achieve more dynamic characterization becomes time and material intensive for compressive strength-based SAI and lime strength tests. Arguably, multiple measurements are easiest to perform for BRI because resistivity is rapid and nondestructive.

6. SUMMARY OF AVAILABLE TESTS

6.1 Materials and Equipment Needed

Table 1 shows the needed materials and equipment for several reactivity tests. The lime strength test is

relatively straightforward because it does not use advanced equipment and uses only a few chemicals. The BRI needs only a bulk resistivity meter, which is becoming more commonly available. The R^3 and modified R^3 tests require relatively complex and expensive equipment unless done using a furnace. While the modified Chapelle test does not need particularly complex equipment, it does need a large number of chemicals, and the procedure can be considered somewhat more challenging than the other tests. Different labs have different equipment, expertise, and so on, and thus, one test is not perfect for all purposes and applications. Generalized, order-of-magnitude cost estimates based on 2024 U.S. dollars are also provided in the table. Costs vary by equipment manufacturer, location, market demand, and other factors and are provided here only for order-of-magnitude comparisons.

Table 1. List of materials and equipment for selected direct and indirect reactivity tests.

| Test | Materials | Major Equipment (Cost) | Other Requirements |
|---------------------|---|--|---|
| SAI test | SCM, cement, and standard sand | Flow table, universal testing machine and compressive strength tester (generally \$40,000)* | Distilled water, mixing equipment, and curing chamber. |
| R^3 test | SCM, Ca(OH)_2 , KOH, K_2SO_4 , CaCO_3 , and deionized water | Isothermal calorimeter (generally \$80,000)* Oven and muffle furnace (generally \$5,000 each)* | Mixing equipment, calorimeter ampoules, and other accessories. Mixing equipment, sieve (2 mm), desiccator, and balance. |
| BRI test | SCM, cement, and standard sand | Oven and bulk resistivity meter (generally \$10,000)* | Mixing equipment and curing chamber. |
| Modified R^3 test | SCM, Ca(OH)_2 , and KOH | Isothermal calorimetry and TGA (generally \$80,000 each)* Oven and muffle furnace (generally \$5,000 each)* | Mixing equipment, calorimeter ampoules, other accessories, and TGA accessories. Mixing equipment, sieve (2 mm), desiccator, and balance. |

Table 1. List of materials and equipment for selected direct and indirect reactivity tests. (Continued)

| Test | Materials | Major Equipment (Cost) | Other Requirements |
|------------------------|--|--|---|
| Lime strength test | SCM, Ca(OH) ₂ , KOH, K ₂ SO ₄ , CaCO ₃ , standard sand, and high-range water reducer | Oven and universal testing machine and compressive strength tester (generally \$40,000)* | Mixing equipment. |
| Modified Chapelle test | SCM, Ca(OH) ₂ , NaOH, HCl, potassium phthalate, phenolphthalein, and sugar | Furnace and alumina crucible (generally \$10,000)* | Erlenmeyer flasks, burette, pipette, filter paper, balance, graduated cylinder, volumetric flasks, stir bars, distillation column, and hot plate. |

*These costs are provided for presenting a general order of magnitude only in the year of publication and may not be representative of quoted costs.

6.2 How To Classify Materials Based on Reactivity

The discussed tests have proposed various classifications for reactivity, except for the Chapelle and Frattini tests. Generally, the reactivity classifications include a threshold below which the material is considered an inert filler and a transition between reactive and highly reactive materials. Because the modified R^3 test also differentiates latent hydraulic and pozzolanic material, an additional form of classification is possible in that test. Classification and thresholds are inherently subjective because reactivity is not a discrete but a continuously varying parameter. Such threshold subjectivity can be easiest understood by considering blends of LS (20 J/g SCM heat release in the R^3 test) and metakaolin (1,100 J/g SCM). By incrementally varying the blend constituents, every value from 20 to 1,100 J/g SCM can be obtained, and a threshold of 98 J/g SCM is inherently as subjective as one of

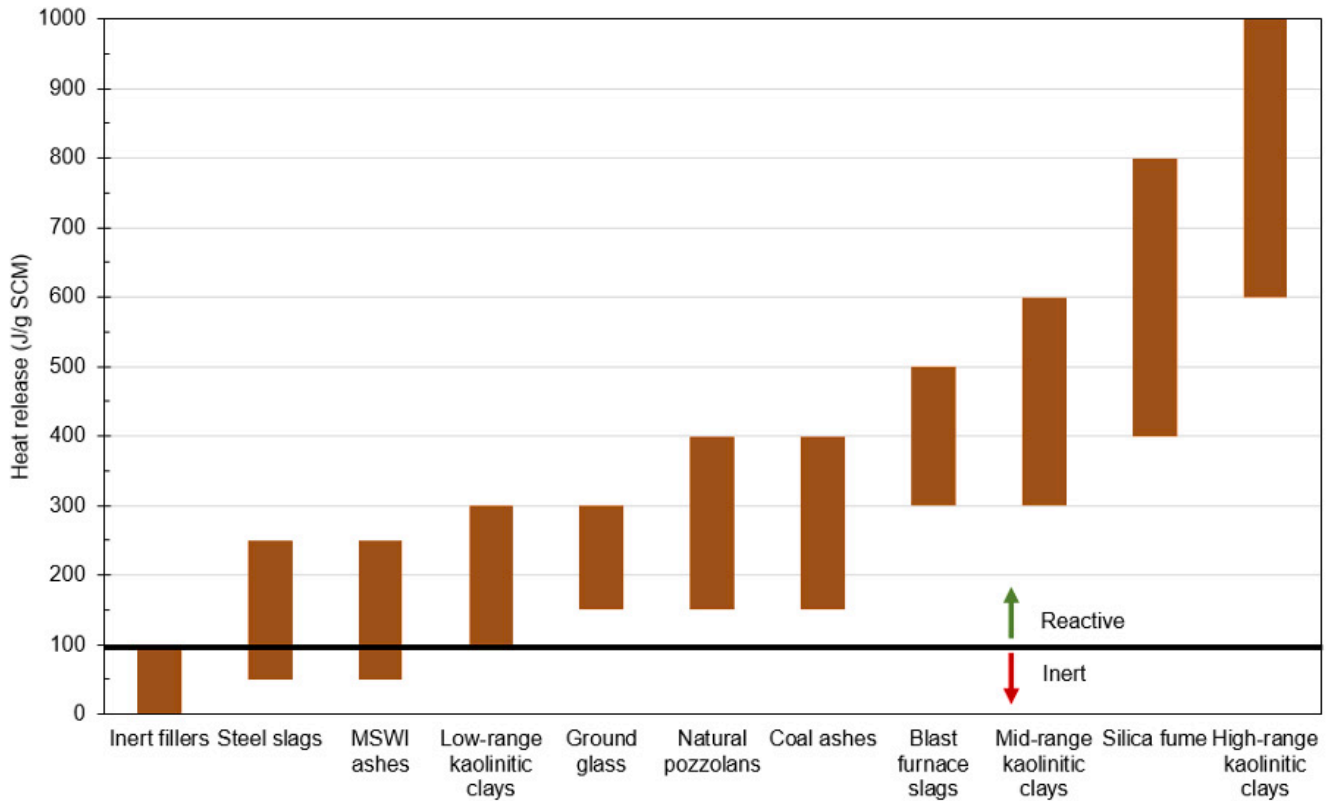
100 J/g SCM. Therefore, if a test result is ambiguous in its classification as inert or reactive, further testing may be desired to validate the performance (e.g., strength and ASR). Table 2 provides the typical classifications in the most common reactivity tests.

In all tests, metakaolin (and other, higher kaolin CCs) and SF are the most reactive, followed by slags, natural pozzolans, and fly ash, with inert materials such as LS showing the lowest reactivity. However, the fact that slag reacts in a manner different from the other materials (latent hydraulic compared with pozzolanic) is apparent only when two measures of reactivity are used, although the different reaction is expected based on compositional differences. Figure 9 displays the typical ranges of heat released by various SCMs and how those ranges compare with the reactive versus inert thresholds presented.

Table 2. Classification in common direct and indirect reactivity tests.

| Test | Classification mode | Classification |
|----------------------|--|--|
| SAI test | Based on strength activity at 7 or 28 d (ASTM C618) ⁽⁹⁾ | Inert: SAI <75 percent at 7 and 28 d. SCM: SAI >75 percent at 7 or 28 d. |
| R^3 test | Based on heat release (J/g SCM) or bound water (g/100 g dried paste) ⁽³²⁾ | At 66-percent confidence level. Inert: Heat release <98 J/g SCM (method A). Inert: Bound water <3.6 g/100 g dried paste (method B). SCM: Heat release >98 J/g SCM (method A). SCM: Bound water >3.6 g/100 g dried paste (method B). At 90-percent confidence level. Inert: Heat release <160 J/g SCM (method A). Inert: Bound water <4.5 g/100 g dried paste (method B). SCM: Heat release >160 J/g SCM (method A). SCM: Bound water >4.5 g/100 g dried paste (method B). |
| BRI test | Based on BRI at 7 or 28 d ⁽⁴¹⁾ | Inert: BRI <100 percent at 7 and 28 d. SCM: BRI >100 percent at 7 or 28 d. |
| Modified R^3 test | Based on heat release and CH consumption at 10 d ⁽³⁵⁾ Based on heat release at 3 d (adapted from reference 35 based on kinetics) | Inert: CH consumption <50 g/100 g SCM, heat release <120 J/g SCM. Pozzolanic, less reactive: 40 g/100 g SCM < CH consumption <100 g/100 g SCM, 200 J/g SCM < heat release <370 J/g SCM. Pozzolanic, more reactive: CH consumption > 100 g/100 g SCM, heat release >400 J/g SCM. Latent hydraulic, more reactive: 20 g/100 g SCM < CH consumption <50 g/100 g SCM, 370 J/g SCM < heat release <550 J/g SCM. Inert: Heat release <100 J/g SCM. SCM: Heat release >100 J/g SCM. |
| Lime reactivity test | Based on 7-d strength ⁽⁴⁹⁾ | Inert: Strength <3 MPa. Low reactivity: 3 MPa < strength <5 MPa. Moderate reactivity: 5 MPa < strength <20 MPa. High reactivity: 10 MPa < strength <20 MPa. Very high reactivity: strength >20 MPa. |

Figure 9. Graph. Typical reactivity of various types of SCMs.



Source: FHWA.
MSWI = municipal solid waste incineration.

Reactivity measurements such as the R^3 test can be incorporated into ASTM standards such as ASTM C618⁽⁹⁾ by using the limits in table 2. WK70466, the new SCM specification currently being balloted,⁽⁵¹⁾ requires that SCMs show at least 90 J/g SCM heat release at 7 d or 3.5 g/100 g dry paste bound water and requires that SAI requirements be met. While reactivity tests such as the R^3 test are more reliable than the SAI test, they are not yet widely implemented in the industry. The easiest-to-implement test, which is likely the R^3 bound water test, could possibly be added as a report-only item to build confidence in the tests that will ultimately replace the SAI test in future years. Reactivity test values alone are not adequate measures of material performance, and thus, reactivity should not be used to rank materials—especially those with different physicochemical properties.

Because the reactivity tests do not use cements, a material shown failing those tests (e.g., having heat release below 98 J/g SCM) is likely to be a highly crystalline material, such as a steel slag, which is not expected to contribute in a significantly positive manner to concrete durability. Technically, such material is likely a filler and should be specified under ASTM C1797⁽¹⁹⁾ and not under ASTM C618⁽⁹⁾ or other SCM specifications. Filler materials can be used in concrete, and LS is, arguably, the most used cement replacement. However, given their low reactivity, specifying fillers as SCMs is likely to be misleading. Such inert materials could be blended with SCMs or activated using various processing methods.^(52,53) Inert materials should be used in concrete with the understanding that such materials will not contribute significantly to improving concrete durability.

Table 3 shows the advantages and disadvantages of various reactivity tests. The tests in table 3 are not pozzolanicity tests; they are overall reactivity tests unless a measure of calcium hydroxide consumption is added to them. Recent discussions have involved an ultra-low-cost reactivity test that uses penetration resistance of R^3 mixtures. When performed in conditions similar to the R^3

test (using the same temperature and pore solution) and benchmarked against R^3 heat release values, researchers can possibly differentiate inert materials from SCMs based on the rate of hardening of the mixtures. Based on the kinetics and extent of hardening, information about kinetics and total reactivity could also be obtained.

Table 3. Advantages and disadvantages of different direct and indirect reactivity tests.

| Test | Advantages | Disadvantages |
|--------------------------------------|---|--|
| SAI test | <ul style="list-style-type: none"> • Test is cost effective and user friendly because equipment and skills needed to perform testing are common for concrete labs. • Equipment is available in most labs. • Test is standardized. | <ul style="list-style-type: none"> • The strength activity threshold is low, resulting in materials with lower reactivity being classified as SCMs. • Interpretation challenges arise due to the requirement for constant mortar flow allowing for the use of variable w/cm. • The low replacement level of SCM may not adequately reflect materials that can be used at higher replacement levels. • High variability in the strength measurement due to the variable w/cm results in interpretation challenges since strength and water content are highly correlated. |
| R^3 test method A: Heat release | <ul style="list-style-type: none"> • The measurements are continuous. • Heat release is linked to strength and durability. • The results are easily interpretable and benchmarkable. • Test is standardized. | <ul style="list-style-type: none"> • Equipment is less commonly found in most labs. • Testing requires skilled and meticulous labor. • More steps are involved for continuous measurements. |
| R^3 test method B: Bound water | <ul style="list-style-type: none"> • Bound water content is linked to strength and durability. • The results are easily interpretable and benchmarkable. • Test is cost effective and user friendly because equipment and skills needed to perform testing are common for concrete labs. • Equipment is available in most labs. • Fewer steps are required to perform method B than for method A. • Test is standardized. | <ul style="list-style-type: none"> • Test is time consuming and laborious to perform. • Measurement is not continuous. |

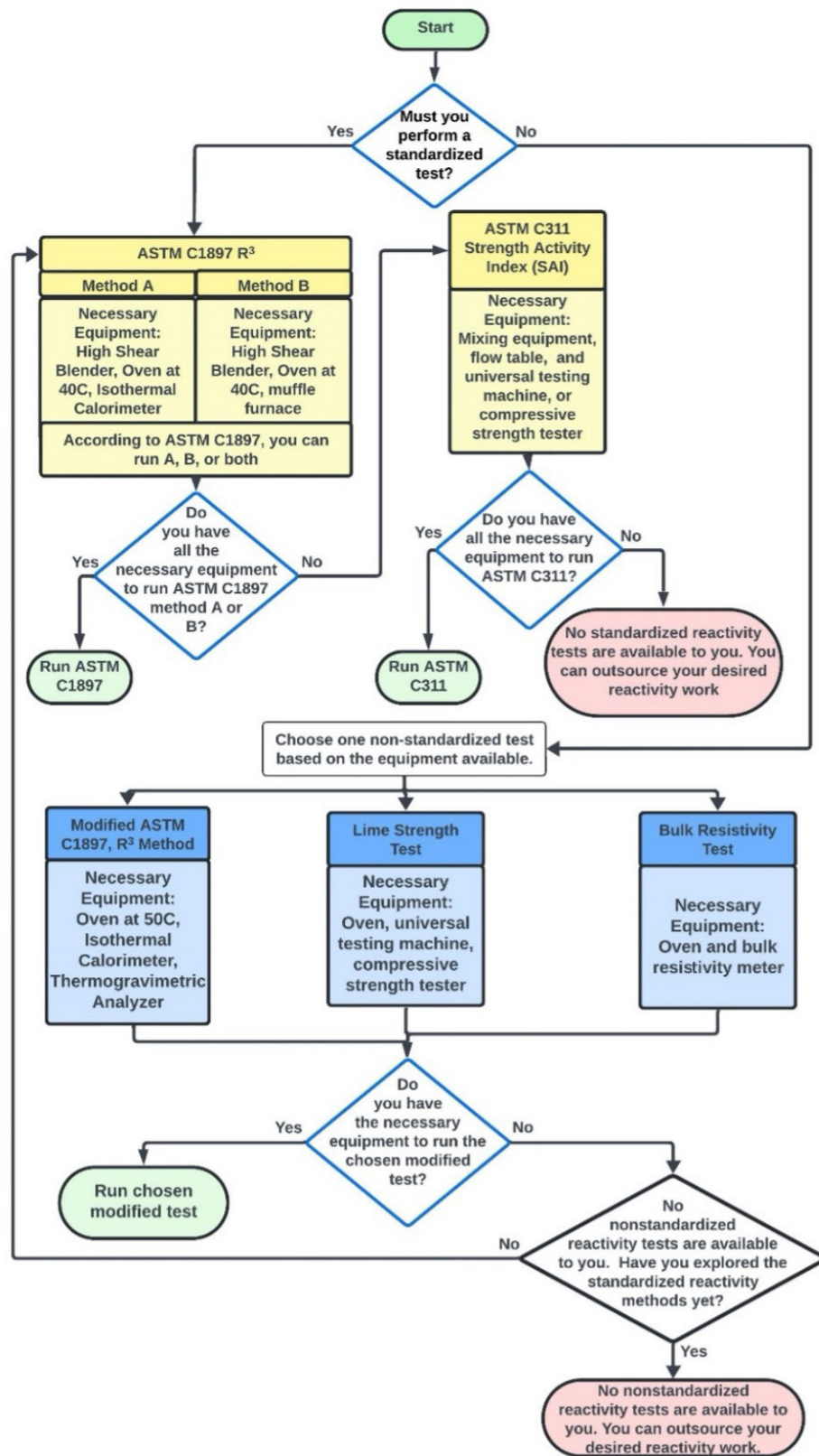
Table 3. Advantages and disadvantages of different direct and indirect reactivity tests. (Continued)

| Test | Advantages | Disadvantages |
|--|--|--|
| BRI test | <ul style="list-style-type: none"> • Test is cost effective and user friendly because equipment and skills needed to perform testing are already available for some concrete labs. • Equipment is available in most labs. • Resistivity is linked to durability. • The results are easily interpretable and benchmarkable. • Resistivity is highly sensitive to reactivity. | <ul style="list-style-type: none"> • Measurement is not continuous. • A lack of data, including round robin and robustness testing, suggests higher uncertainty in the results. • Test is not standardized. |
| Modified R^3 test: Heat release and bound water | <ul style="list-style-type: none"> • Pore solution lacks sulfates and carbonates, reducing pore solution complexity compared with that for the R^3 test. • The same advantages apply for the modified R^3 test as for the R^3 test, except the modified R^3 test is not standardized. | <ul style="list-style-type: none"> • The same disadvantages apply for the modified R^3 test as for the R^3 test. • Test is not standardized. |
| Lime strength test | <ul style="list-style-type: none"> • Test is cost effective and user friendly because it uses generic concrete lab equipment and skills. • Equipment is available in most labs. • The results are easily interpretable and benchmarkable. | <ul style="list-style-type: none"> • Measurement is not continuous. • Test is not standardized. |

6.3 How To Determine Which Reactivity Test To Use

Figure 10 presents a flowchart to facilitate the determination of which reactivity test may be used based on the information desired and the resources available.

Figure 10. Diagram. Flowchart for determining which reactivity test to use.



7. LINKS BETWEEN REACTIVITY AND PERFORMANCE

7.1 How Reactivity Ties to Materials Performance

The original R^3 test was developed as a way to predict the 28-d compressive strength of mortars with SCMs, and subsequent work on that test by the RILEM TC 267-TRM continued to benchmark the test against compressive strength.^(27,32,33) The R^3 test results correlate well with compressive strength for the calcined kaolinitic clays for which the test was developed.⁽²⁷⁾ When the R^3 test results are expanded to a broader class of SCMs, the correlation to 28-d compressive strength is still good but less compelling. Al-Shmaisani et al.,⁽⁷⁾ for example, compared 28-d mortar compressive strength with a variety of SCMs with the 7-d heat release from R^3 testing and found a linear correlation with R^2 equal to 0.79. Researchers can expect scatter in strength measurements because strength is a complex measure affected by other parameters in addition to reactivity. Al-Shmaisani et al.⁽⁷⁾ assessed whether reactivity tests could predict other types of concrete performance, including concrete compressive strength, ASR expansion, sulfate resistance, and rapid chloride penetrability. Since the R^3 test⁽²⁸⁾ is a reactivity test and not a pozzolanicity test, it did not predict performance well unless accompanied by additional chemical composition information about the SCM to suggest a pozzolanic or latent hydraulic reaction.

Other authors have also suggested that caution be exercised in the linking of measures of reactivity to concrete durability.⁽⁶⁾ In one study, the authors showed strong links between pozzolanic reactivity (measured using calcium hydroxide consumption in the modified R^3 test), bulk resistivity, and ASR expansion.⁽⁵⁴⁾ Generally, materials that show higher calcium hydroxide consumptions result in higher bulk resistivity values.⁽⁵⁴⁾ However, resistivity is affected by many other parameters. For example, two fly ashes with similar calcium hydroxide consumption may have different alkali contents or bind alkalis in the C-S-H differently, resulting in concretes with different resistivity values. SCMs can have high free alkalis that result in high ASR expansion but still be pozzolanically reactive, and thus reactivity alone cannot be used to predict durability. However, reactivity testing, such as via the R^3 or modified R^3 test, can be used for preliminary screening to identify low-reactive materials that would not assist in mitigating ASR expansion. Additional work is needed in the use of reactivity tests to predict concrete performance. Using reactivity to predict concrete performance must be done

with caution and may be best suited to identify materials that would not help with concrete durability. Such identification can help reduce extensive durability testing.

7.2 Additional Testing That Could Serve To Increase Confidence in Using the Material

As pointed out in section 2.2, reactivity is, arguably, the single most important information that can be obtained about an SCM. However, just because an SCM is reactive based on meeting some threshold (e.g., 200 J/g SCM 7-d heat release in the R^3 test) does not mean the SCM can be broadly used in concrete. Certain other factors are important:

- **Chemical composition:** X-ray fluorescence (XRF) should be performed on the material. Ideally, alkali contents, loss on ignition, chloride contents, organics, sulfates, and deleterious elements and oxides should be low.
- **Mineralogy:** Ideally, x-ray diffraction (XRD) should be performed on the material. However, it is well known that XRD on cementitious materials is complex. XRD can help quantify the contents of crystalline and amorphous phases and detect the presence of deleterious phases harmful to hydration.
- **Particle-size distribution:** Some measure of particle-size distribution or specific surface area is needed for SCMs. Typical SCMs have median particle sizes around 15 μm . If the particle sizes are much coarser, reactivity is likely to be limited. On the other hand, when they are much finer, water demand of the SCM is likely to be a major issue. In some cases, for materials such as zeolites, which have complex internal surfaces and porosities, surface area may also be relevant to examine in addition to particle-size distribution.
- **Hydration:** Isothermal calorimetry on the cement paste containing reasonable amounts of SCMs should be performed, if possible. Abnormal hydration behaviors, such as rapid or slow setting and sulfate imbalance, can be causes for concern, although why they occur may be hard to identify without XRD information.
- **Strength and bulk resistivity:** Strength gain and bulk resistivity of cement mortars or concretes containing reasonable amounts of SCMs should be measured at 1, 7, 28, 56, and 90 d. Abnormal strength gain behavior should be noted.

In principle, composition (XRF, XRD), particle size, hydration, strength, and bulk resistivity in combination with reactivity offer a reasonable understanding of a given SCM and how it would affect concrete. If a material shows adequate reactivity, has no or limited amounts of deleterious phases, has appropriate particle size, and shows satisfactory hydration, strength gain, and bulk resistivity, then it is an SCM that may be considered for use in concrete and will likely contribute positively to durability.

7.3 How Reactivity Ties to Materials Resource Efficiency

Reducing the amount of clinker in cement and the amount of cement in concrete can improve the concrete industry's cost and resource efficiency. One of the most straightforward ways to reduce cement content is to replace it with SCMs. Reactivity testing can increase confidence in new products and help the products gain market acceptance. Thus, reactivity testing can help increase the amounts of SCMs used to replace cement and potentially increase SCM availability around the United States. Materials that show very high reactivity can typically be used only at low replacement levels due to cost and water demand, and materials that show low reactivity can be used at moderate replacement levels due to strength reductions. Thus, materials that show moderate to high reactivity, such as slags, typically can be used at high replacement levels. A better understanding of the link between reactivity and cement replacement levels can help the industry develop designer SCMs that can be used at high replacements while minimizing effects on strength and water demand, improving concrete performance while utilizing local materials.

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