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Reaction kinetics of supplementary cementitious materials in reactivity tests

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ABSTRACT

This work characterizes the reaction kinetics of supplementary cementitious materials (SCMs) with calcium hydroxide in the modified R^3 test. The heat flow curves of 58 SCMs of varying reactivities were studied. Based on the heat flow curves, the SCMs were classified as more reactive, less reactive, and inert. Most of the heat flow curves in the modified R^3 test exhibit, after the peak of heat flow, an initial slow decaying power-law regime that transitions into a longer and faster decaying power-law regime. The pre-exponent of the first regime depends on the initial SCM reactivity and correlates well with the 24-hour heat release in the modified R^3 test, thus making it a useful metric for rapid classification of SCMs.

1. Introduction

The use of supplementary cementitious materials (SCMs) as partial replacement to ordinary portland cement (OPC) in concrete reduces CO_2 emissions associated with cement manufacture and generally improves concrete durability [1–4]. The availability of conventional SCMs, such as fly ash, has decreased due to the phasing out of the relevant industries (e.g., coal power plants) because of environmental and cost concerns. Therefore, there is a pressing need to use alternative or novel SCMs in concrete. However, to effectively use novel SCMs, their reactivity needs to be assessed and understood. Reactivity tests such as the strength activity index test, the Chapelle test, or the Frattini test have been used for years for conventional and alternative SCMs but have critical flaws [5–8]. As an example, for the strength activity index test, the variable water-to-cementitious materials ratio and the early age of testing obscure the contribution of the SCM reaction, and consequently lead to potential misclassification of inert materials [8]. The Chapelle and Frattini tests are based on portlandite consumption, and thus are not truly applicable to latent hydraulic materials such as slag [5–7].

Recent advances in reactivity testing have yielded four promising SCM reactivity tests – the bulk resistivity index test [8], modified lime strength test [9], the R^3 test [10], and the modified R^3 test [11], which overcome most of the shortcomings of previous tests. Therefore, these tests can be used to classify SCMs based on their reactivity [12]. The R^3 test measures 7-day heat release and/or bound water content in model systems containing SCM and calcium hydroxide in a high-pH solution at 40°C with added sulfates and carbonates [10,13]. The R^3 test is reflective of 28-day behavior, as it is based on comparisons with 28-day strength data [13]. However, it may not be well-suited for slowly reacting materials such as Class F fly ash and other siliceous materials, which show limited extent of reactivity at 7 and 28 days [8]. In addition, using heat release or bound water does not easily allow the differentiation of latent hydraulic and pozzolanic materials, which may show similar values of heat release while having very different effects in concrete [11]. The modified R^3 test [11] measures the 10-day heat release and calcium hydroxide consumption in similar model systems at 50°C but without added sulfates and carbonates. Removing the extra sulfates and carbonates simplifies the system. The exact roles of sulfates and carbonates in the reaction are complex and vary depending on whether the SCMs are largely siliceous, aluminosilicates, or calcium aluminosilicates in terms of their composition [14]. The higher temperature and longer duration make this test more suitable for slowly reacting materials, and since this test is highly accelerated, its outputs are typically reflective of the behavior of SCMs in cement pastes (or concrete) at later ages (beyond 56 days) [15,16]. By measuring both heat release and calcium hydroxide consumption, the modified R^3 test can be used to classify SCMs into *inert* (IN), *pozzolanic, less reactive* (P, LR), *pozzolanic, more reactive* (P, MR), *latent hydraulic, less reactive* (LH, LR), and *latent hydraulic, more reactive* (LH, MR) categories [12]. This ability for classification of SCMs is a critical advantage with respect to

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other tests.

Early-age measurements of heat release in reactivity tests, which could provide important insight into the behavior of cementitious mixtures, have not been fully explored. While isothermal calorimetry offers a continuous measurement of heat release, only the heat release at the end of the test duration is typically used as a measure of reactivity. Understanding the reaction kinetics in the modified R^3 test and other reactivity tests is critical to obtain a full picture of SCM reactivity and to correlate SCM behavior in model systems and cementitious paste properties [17].

Analytical models used to describe the hydration kinetics of cementitious systems, such as C_3S mixed with water [18], generally rely on Avrami-type nucleation and growth formulations which assume an exponential or stretched-exponential decay in the heat flow after the peak [19]. Power law fits, arising from the complex interplay of dissolution, nucleation and growth, and diffusion phenomena, have also been suggested [18]. More sophisticated numerical methods, such as thermodynamic modeling or the Parrot and Killoh model, have also been used to study hydration of OPC and SCM-OPC/C_3S systems [20–26]. Other methods like ab initio calculations, kinetic Monte Carlo simulations or mathematical models of dissolution, nucleation, and growth, have provided insight into different aspects of the hydration process [18, 19,27–29]. Whether classical hydration kinetic models apply to pure SCM model systems (with calcium hydroxide at high pH), remains unclear.

In this study, for the first time, the reaction kinetics in the modified R^3 test are characterized and modeled. Specifically, this is done on 58 SCMs with varying reactivities which were tested for a duration of 10 days. The potential of using the heat flow curves and fitting parameters for rapid SCM classification is discussed.

2. Materials and methods

A total of 58 SCMs of varying levels of reactivity and different classifications – inert, pozzolanic, and latent hydraulic, were evaluated in this study. The SCMs include 4 basalt fines (BF1-4), 7 biomass ashes (BMA1-7), 4 calcined clays (CC1-4), 12 Class C fly ashes (CFA1-12), 12 Class F fly ashes (FFA1-12), 2 glass powder pozzolans (GP1-2), 2 silica fumes (SF1-2), 5 ground granulated blast furnace slags (SL1-5), 2 pumices (P1-2), 2 limestone powders (LS1-2), 1 each of nepheline syenite filler (NF), ground bottom ash (GBA), sandstone powder (SST), quartz (Q), ladle furnace slag (LFS), and a processed silica SCM (SP). The chemical compositions of these SCMs (from x-ray fluorescence) and d_{50} values (from laser diffraction), where available, are provided in Table A1 (Supplementary Material). More details regarding BF1 can be found in [12], BMA6-7 in [30,31], MK1-3 in [12,32], GP1-2 in [12,15], SF1-2 in [15,16], SL1-5 in [12], P1-2 in [15], LS1-2 in [16], LFS in [33], and, BMA1-5, and NF, GBA, SST, and SP in [34]. While reactivity data for some materials has been published, reaction kinetics and their modeling have not been previously investigated.

2.1. Modified R^3 test

The modified R^3 test was carried out on all SCMs based on the method described in Suraneni and Weiss [11]. Reagent grade calcium hydroxide and the SCM (mass ratio 3:1) were dry mixed together for four minutes in a plastic container to ensure uniform distribution. A freshly prepared 0.5 M potassium hydroxide solution was added to the dry powder mixture at a liquid-to-solid ratio of 0.9. Because of the small quantities of components involved, they were hand mixed thoroughly for four minutes. From this mixture, 6-7 g were placed in a glass ampoule, sealed, and placed into an isothermal calorimeter (TAM Air, TA Instruments) preconditioned at 50 ± 0.05°C. The heat flow data for the first 45 minutes was not collected due to temperature differentials. The heat release parameters were collected for 10 days. At the end of this period, thermogravimetric analysis (TGA55, TA Instruments) was performed using approximately 30-50 mg of the sample obtained from the bulk. The temperature was ramped at 10°C/minute from ambient to 600°C in an inert nitrogen atmosphere. The mass loss in the range of approximately 380-460°C was measured using the tangential method described in Kim and Olek [35] and used to determine calcium hydroxide content, from which calcium hydroxide consumption was calculated. For selected specimens, TGA was carried out up to 1000°C to check for carbonation; significant carbonation was not detected. TGA was performed within 12 hours of completing the isothermal calorimetry measurements and within 15 minutes of breaking the ampoule. Replicate testing of five randomly chosen SCMs showed that the differences in heat release and calcium hydroxide consumption values between specimens were less than 5%. The SCMs were classified using previously established criteria [12,16]. The classification results are shown in Fig. A1 (Supplementary Material). Based on the classification, BF, LS, NF, SST, LFS and Q were classified as inert; P, GP, BMA1, BMA6-7, FFA, CFA were classified as pozzolanic, less reactive; SF, CC, BMA2-5, SP, and GBA were classified as pozzolanic, more reactive; SL was classified as latent hydraulic, more reactive. Five of the 58 SCMs could not be unequivocally assigned to any classes, which highlights a limitation of the classification criteria. Nonetheless, those SCMs were assigned to the class to which their distance to the classification boundary was the least.

3. Results and discussion

Fig. 1 shows the heat release curves of example Class C and Class F fly ashes. Up to 3 days, the Class C fly ash was significantly more reactive than the Class F fly ash, with heat release values being up to 50% higher. The Class F fly ash reacted in a slower but more sustained manner, and at the end of the test (10 days), the difference in the heat release values of the two materials was insignificant (5%).

Numerous Class C and Class F fly ashes have been studied in our group using the modified R^3 test [12], and this behavior always appeared in the heat release response. The reactivity of the fly ashes in the modified R^3 test was consistent with their reactivity in cementitious pastes (albeit at different time scales due to the accelerated nature of the modified R^3 test). These differences in reactivity are driven by differences in the reactivities of the siliceous, aluminosilicate, and calcium aluminosilicate glasses that comprise the fly ashes [14]. It is critical to understand the reaction kinetics; otherwise, incorrect assessments of material reactivity could be made.

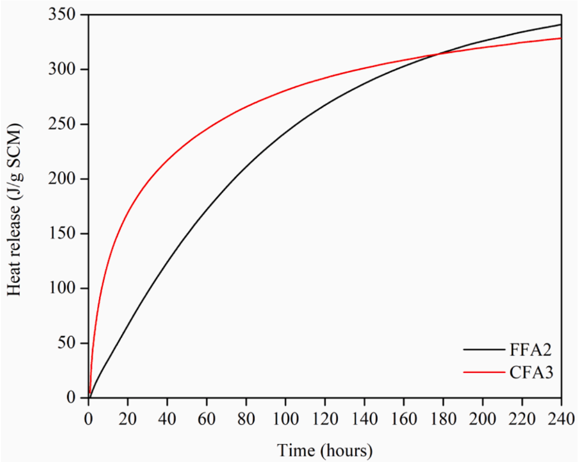

Fig. 1. Heat release curves of example Class C (CFA3) and Class F (FFA2) fly ashes.

3.1. Modeling the heat flow curves and prototypical heat flow curves for different classes of SCM

The data for the first 45 minutes to one hour after mixing in the modified R^3 test were not collected due to temperature differentials. Generally, the first peak in the rate of heat release occurred within this time interval and thus was not measured for most of the SCMs studied. However, for nine slower reacting SCMs, the heat flow before and leading to the peak value was captured. Because here we only focus on modeling the post-peak evolution of the heat flow (i.e., the derivative of the heat release), the aforementioned nine SCMs were not modeled for consistency.

The reaction of the SCMs and calcium hydroxide in the modified R^3 test is a complex process and the reaction rate depends on factors including but not limited to the glass content, the chemical composition of the calcium aluminosilicate glass, activation energy, and SCM fineness [36]. Interestingly, the 240-hour heat flow curves of most of the non-inert SCMs studied here could be best captured by two power-law regimes (Fig. 2): a shorter, slower decaying regime (Eq. 1) that transitioned at time t_{th} into another longer, faster decaying one (Eq. 2). The reaction kinetics according to this model are captured by four parameters A, B, and α, β which correspond to the pre-exponential factors (i.e., the intercepts at t = 1 hour) and the exponents (i.e., the slope in a log-log scale), of each of the regimes respectively:

$$Q = At^{-\alpha} \quad \text{when } t < t_{th} \tag{1}$$

$$Q = Bt^{-\beta} \quad \text{when } t > t_{th} \tag{2}$$

where Q and t are heat flow (in mW/g SCM) and time (in hours).

The pre-exponent A is the intercept of the fit with the y-axis at t = 1 hour, and is thus an indicator of the early heat flow. The exponents α and β capture how fast the heat flow decayed over time, with larger values implying faster decays. To calculate the parameters of the model, the initial part of the curve (0-4 hours) and the tail of the curve (61-240 hours) were fit to respective power-laws. The threshold time, t_{th} (which is not a fitting parameter), was determined as the time where the two power-laws intersected. It is worth noting that the values of the parameters were not sensitive to the range of time scales used to fit the two regimes. For example, the differences in the pre-exponent values were less than 15% when using 0-6 hours and 39-240 hours to fit the first and second regimes respectively.

The heat flow curves from the modified R^3 test generally fell into three prototypical curve shapes, which we refer to as type MR (more reactive), LR (less reactive), and IN (inert) (Fig. 3). In 16 out of the 58 SCMs studied here, the t_{th} values were either less than t = 1 hour or more than t = 240 hours. In such cases, mostly corresponding to IN or MR curves, the heat flow was modeled using a single power-law ($Q = At^{-\alpha}$).

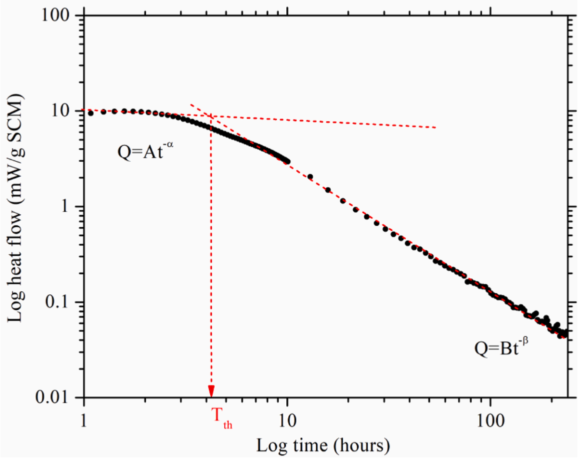

Fig. 2. Fitting procedure and calculation of threshold time.

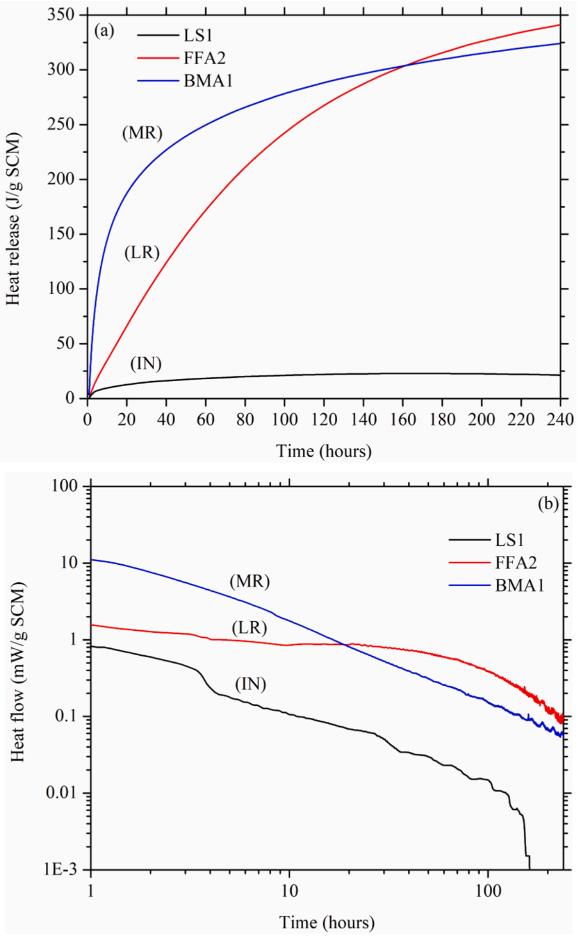

Fig. 3. Typical curves for SCMs in the modified R^3 test – (a) Heat release and (b) Heat flow (log-log).

For MR SCMs, the parameter A, related to the initial heat flow, was between 10-40 mW/g SCM. The heat release curves for the MR SCMs showed a rapid initial increase in the heat release, with a slower increase at later ages. SCMs such as silica fume, metakaolin/calcined clays, Class C fly ashes, and blast furnace slags followed this type of curve. These materials are either aluminosilicates, calcium aluminosilicates, or fine siliceous materials.

The LR SCMs had a much lower initial heat flow (A in the range of 1-5 mW/g SCM), which decayed extremely slowly until about 20-100 hours (the t_{th}), and then more rapidly until reaching similar values of heat flow as the MR SCMs at later times. Initially, the heat flow of the MR SCMs was higher than for LR SCMs; but a crossover was observed at later times . In the heat release plot, Type LR curves showed a gradual increase in the heat release for 20-100 hours, but the rate of change decreased with time. Examples of LR SCMs include Class F fly ashes, pumice, and glass powder – all siliceous materials. The siliceous materials do not necessarily show low values of heat release (Fig. 1) and are perhaps better termed as slowly reactive materials [14].

The IN SCMs are characterized by low initial heat flow values (1 mW/g SCM or below), and typically exhibited just a single power-law regime. A sharp decrease in the heat flow at around 200 hours was seen for IN SCMs, although it was unclear whether this drop was an experimental artefact due to the low heat flow values which made the response indistinguishable from noise [11,12]. The final heat flow values for IN SCMs were significantly lower than either MR or LR SCMs. Examples of IN SCMs include limestone and quartz. IN SCMs showed a low heat release through the duration of the experiment. The fitting parameters for all SCMs in this work are provided in Table A2 (Supplementary Material).

The classification of SCMs as MR, LR, or IN based on their kinetics (i.e., the phenomenology of the heat flow curves), are not necessarily related to the more fine grained classification (IN; P, LR; P, MR; LH, LR; and LH, MR) based on the 10-day value of the heat release and calcium hydroxide consumption, which is an indicator of overall reaction and type of reaction [12].

3.2. Model parameters and different SCM classes

The values of the fit parameters of the kinetic model were investigated for different SCM classes. Fig. 4 shows for different SCM classes the values of the pre-exponent A (Fig. 4a), which is related to the initial heat flow, the exponent α (Fig. 4b), which relates to how fast the heat flow decreases over time, and the threshold time t_{th} (Fig. 4c), at which the reaction kinetics transition from a slowly decaying regime to fast decaying one. The range of these values for all SCM classes are also shown in Table A3 (Supplementary Material).

The value of A was one or two orders of magnitude greater for reactive materials than for the inert materials. The fact that differences in SCM behavior could be identified using model parameters that can be determined using just the first few hours of testing is significant as test times could be substantially reduced. While the test duration can be reduced to 1-day or 3-days, this should not be done for the slowly reacting LR SCMs without careful kinetic corrections [14]. For Class F fly ashes ($\mu = 5$, $\sigma = 5$), A values were significantly lower than that of Class C fly ashes ($\mu = 11$, $\sigma = 3$), where μ and σ are the mean and the standard deviation, respectively.

The values of α did not strongly depend on the SCM class except for IN SCMs. For Class F fly ashes ($\mu = 0.4$, $\sigma = 0.2$), the α values were about 40% smaller than those of Class C fly ashes ($\mu = 0.6$, $\sigma = 0.2$). The range of values for t_{th} for the different SCM classes somewhat overlapped (Fig. 4c); however, there was a significant difference in the average values of P, LR and P, MR SCMs. Threshold times could not be determined for some P, MR SCMs (such as calcined clays and silica fume) due to their rapid reaction and for some P, LR SCMs, as the convergence was beyond 240 hours. The average t_{th} for Class F fly ashes ($\mu = 63$ hours, $\sigma = 42$ hours) was more than twice that of Class C fly ashes ($\mu = 27$ hours, $\sigma = 33$ hours). Our results show that, initially, reaction rate for Class C fly ashes was greater than that for Class F fly ashes, but decreased faster; and at later ages, the reaction rates for Class F fly ashes were higher [8]. This finding highlights the importance of considering the entire heat curves and not just the reactivity at 10 days, especially for slowly reacting materials. The behavior in these model systems was consistent with the behavior seen in concrete for different fly ash classes [8,17,36]. Although SCM behavior in a cementitious system depends on several factors [36], these results indicate a qualitative estimate of reactivity could be obtained based on A, α, and t_{th}.

Fig. 5 shows the relationships between pairs of model parameters, for the different SCM classes shown in different colors Fig. 5.a shows that Class C fly ash (blue triangles) and Class F fly ash (red dots) occupied distinct regions in the t_{th} vs. A log-log plot. Even though both these SCMs could be classified as P, LR, they follow completely different trends, because the P, LR classification did not consider reaction kinetics [8]. The slags (pink diamonds) occupied a distinct region in the plotthan other SCMs, likely because of their latent hydraulic properties [12]. As one moves from left to right of the plot, the SCM reactivity increased in the direction represented by the arrow. More reactive materials were characterized by high A values and earlier t_{th}, whereas less reactive materials had low values of A and later t_{th}. However, for Class C fly ashes, there was no correlation between t_{th} and A.

The semi-log plot of A and α in Fig. 5b shows that there was a correlation between these parameters when all SCMs are considered together. The reactivity of SCMs increased as we move from the right to left of the plot in the direction indicated. Further, Class C fly ashes exhibited intermediate reactivity, as observed in both Fig. 5a and Fig. 5b, where they fell between the more and less reactive SCMs.

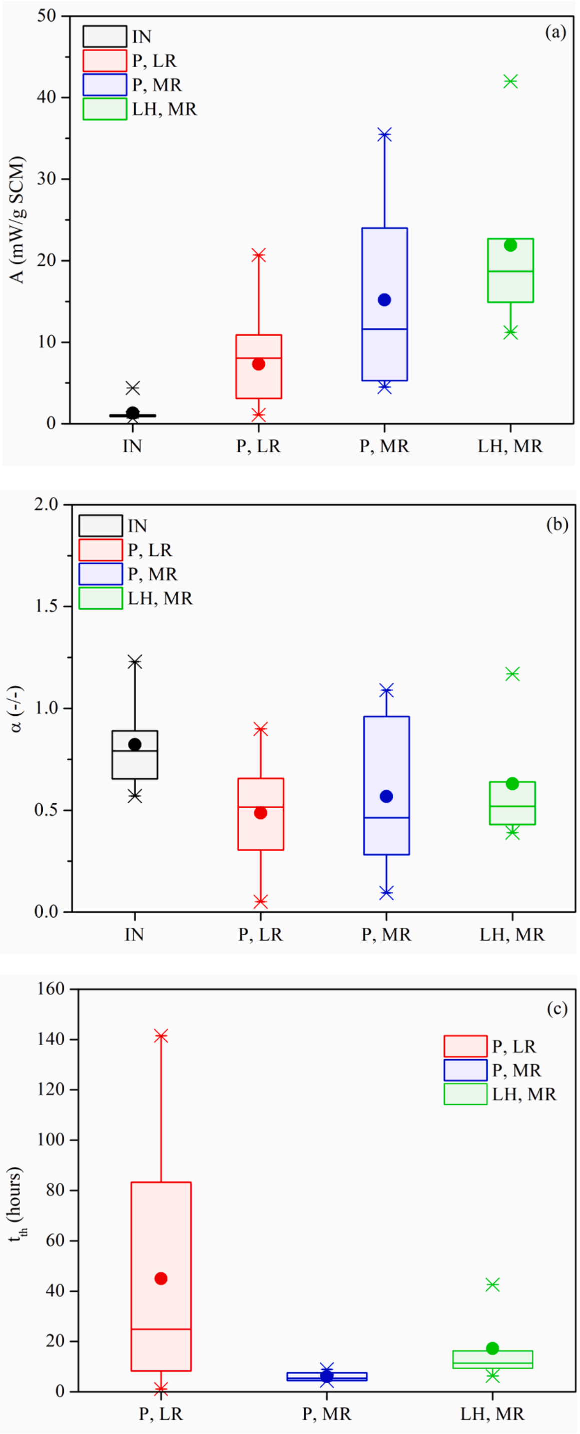

Fig. 4. Range of parameters based on power law model – (a) Pre-exponent A, (b) Exponent α, and (c) Threshold times t_{th}. The crosses on either end of the box plot represent the maximum and minimum values, the top and bottom sides of the box represent the upper and lower quartile values of the dataset, and the dot and line inside the box are the mean and median of the dataset.

Fig. 6a shows the relationship between the 1-day heat release and the values of the parameter A for all the SCMs. There was a moderate, positive correlation between the two parameters. There was clear distinction between inert and reactive SCMs except for two materials. *In principle*, the test duration could be reduced to one day if the objective

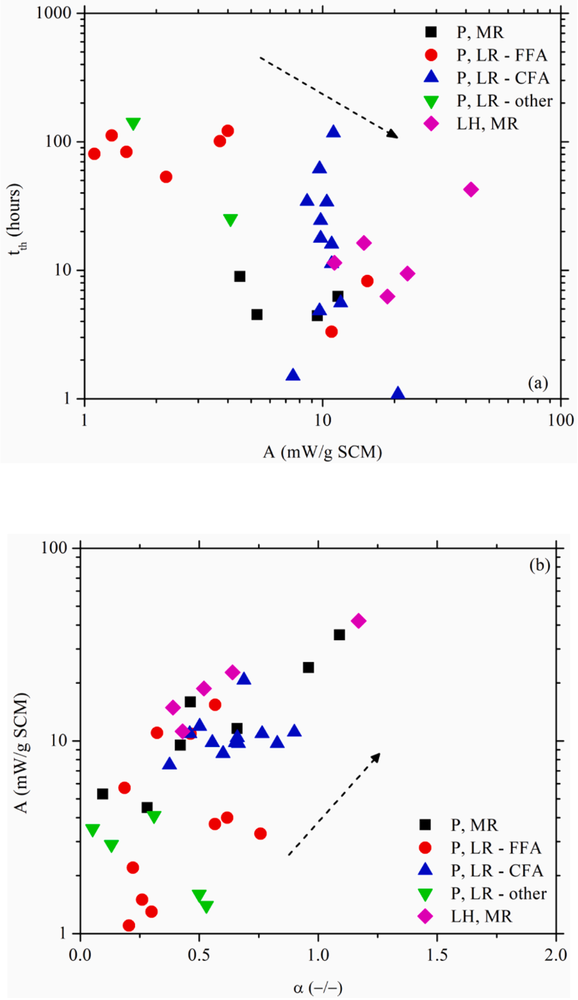

Fig. 5. Two parameter plots for the variables from heat flow curves – (a) t_{th} vs. A and (b) A vs. α. FFA refers to Class F fly ash and CFA to Class C fly ash.

were solely to differentiate inert and reactive materials, which is an important practical consideration [2,8,10] Fig. 6b also shows the moderate correlation between the 3-day heat release and the A value. The separation between inert and reactive SCMs was clearer, as the less reactive Class F fly ashes reacted in the 1-to-3-day period. SCMs of different classes appeared somewhat clustered, which was expected, since only heat release (and not calcium hydroxide) was being used as a reactivity measure [11,12]. At 10 days, while the correlation between the parameters was poorer, there was a clear distinction between reactive and inert SCMs. At all ages, reactivity increased as we move from the right to left of the plot, with some outliers for Class F fly ash, which showed low early age reactivity.

3.3. Practical considerations

Results on a small set of materials suggest that the two-regime kinetic model shown here is also applicable for the R^3 test [37]. Two important, practical findings are worth discussing. The first is that by testing at 50°C, reactive and inert materials could be differentiated as early as 1-day. The demonstrated correlations between different direct and indirect measures of reactivity (heat release, calcium hydroxide consumption, and bound water) from the R^3 and modified R^3 tests [8,11,12,14,37] mean that other tests could also be shortened to 1-day regardless of whether their exact reaction kinetics. While the tests could be shortened to 1-day, it does not appear that doing so will provide a complete picture of the reactivity. Caution should be exercised for Class F fly ashes and other slowly reacting siliceous materials, which show different reaction kinetics. Indeed, research has shown that the 1-day or 3-day heat release in both the R^3 and the modified R^3 test are strong predictors of the 10-day heat release, expect for siliceous SCMs [14].

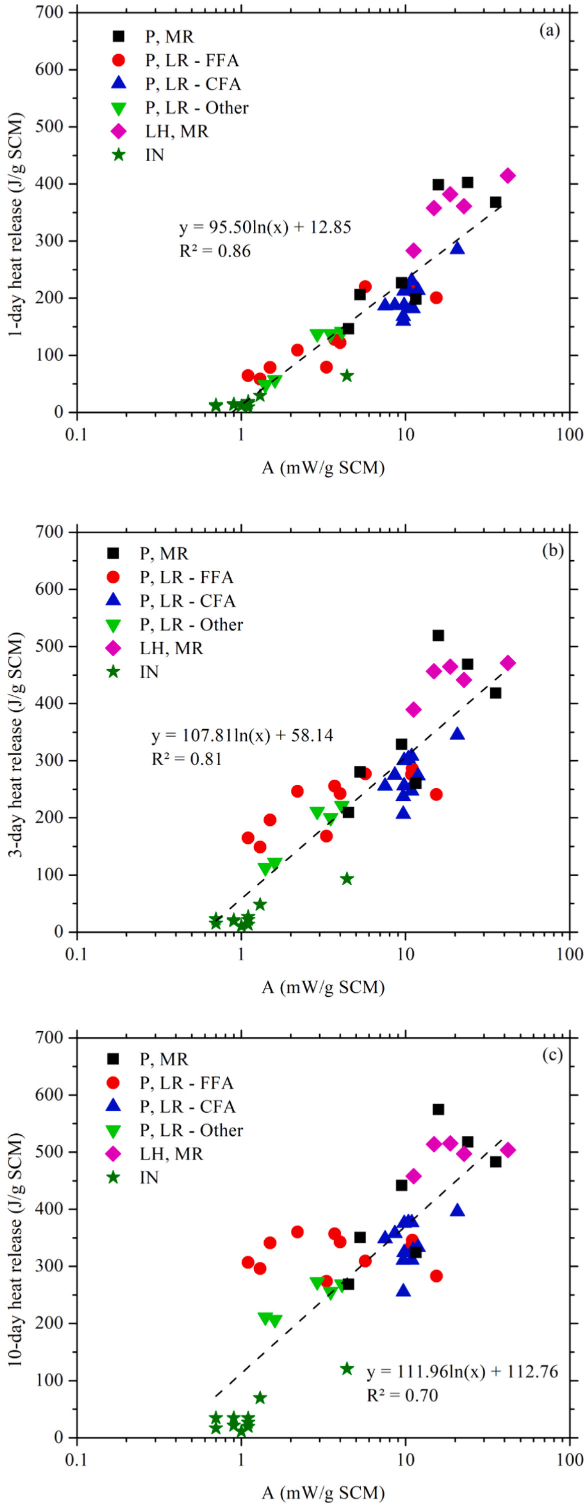

Fig. 6. Two parameter plots of heat release and A value in the modified R^3 test – (a) 1 day, (b) 3 days, and (c) 10 days.

The second important finding is the discovery that the reaction kinetics can be highly variable, even in SCMs showing similar overall reactivity. When discussing reactivity test results, parameters indicative of reaction kinetics should be presented along with those indicative of overall reactivity to provide a better picture of SCM reactivity. A potential, simple, candidate to provide kinetic information would be the ratio of 3-day and 1-day heat release. This parameter averaged 1.94 for the Class F fly ashes and 1.36 for the Class C fly ashes and generally the highest values of this parameter were found for siliceous SCMs. Therefore, by stopping the test at 3-days and using the 3-day and 1-day heat release ratio, a more complete picture of reactivity and reaction kinetics could be rapidly obtained. Extrapolations and curve fitting the heat release could also be used to estimate the 10-day or even ultimate heat release.

The complexity of the kinetics is important to consider when using the single point bound water measurements, as is suggested in the R^3 test (ASTM C1897). While the bound water values will likely distinguish between inert and reactive materials, kinetic information cannot be obtained from them. It could be advantageous to use bound water measurements at two times (1-day and 3-days), especially for siliceous materials.

4. Conclusions

Reaction kinetics of SCMs in the modified R^3 test were evaluated in this study. The major conclusions from this study are –

1. Three prototypical curves were observed to describe the SCM behavior. These curves differ in the initial value of heat flow and the initial and final slope of the heat flow.
2. The reactivity of SCMs according to the modified R^3 test can generally be captured by a kinetic model consisting of two power law regimes. The parameters describing the power law fits, A, α, and t_{th}, provide information about SCM reactivity and can be used for their classification, based on initial reactivity and rate of decay of the heat flow. The parameter A is particularly useful, as it allows for discerning between reactive and inert SCMs potentially within one day only.
3. SCMs with similar long-term overall reactivity can show widely different reaction kinetics, meaning that kinetic information must be considered when discussing SCM reactivity, a finding which has important practical implications.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cement.2022.100022.

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