

Pozzolanic Activity of Rice Husk Ash : Comparison of Various Electrical Methods

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Abstract

The great abundance of rice husk ash (RHA) as low-cost agricultural waste in Thailand makes it the most suitable material for cement-based applications. However, the use of RHA as a supplementary cementitious material (SCM) is not yet widespread in Thailand due to the poor pozzolanic activity of most agricultural RHA. Most RHA is incinerated at too high temperatures (usually in excess of 1000°C) or under uncontrolled conditions as biomass fuel. This reduces the amount of amorphous SiO₂ and hence worsens the pozzolanic activity of the resulting RHA. To enhance the use of RHA as an SCM in Thailand, a proper method to evaluate its pozzolanic activity is necessary. In this work, the two most simple electrical methods proposed in the literature to evaluate the pozzolanic activity of different pozzolans (not necessarily RHA) will be applied to RHA. Their applicability is discussed in comparison with the strength activity index method, and other physical properties believed to influence the pozzolanic activity of RHA.

Key words: Rice husk ash, Impedance spectroscopy, Pozzolanic activity, Electrical conductivity

Introduction

The world's energy crisis has caused many agro-industries in Thailand to use their agricultural wastes as alternative energy sources in place of natural gas and coal nowadays. With 5-6 million tons of rice husk available as biomass fuel each year, this results in approximately one million tons of rice husk ash (RHA) being discarded in land fill, causing environmental and health-related problems to nearby communities. These problems can be alleviated if this RHA can be used, by a large volume, as a supplementary cementitious material (SCM). It will also help reduce the use and production of Portland cement, which is infamous for its enormous energy consumption (1800 kJ/kg) and CO₂ emission (1 kg CO₂/kg) at the same time. To act as a good SCM, the RHA must possess a pozzolanic property, i.e., providing excess reactive SiO₂ to react with Ca(OH)₂ released from cement hydration to yield calcium silicate hydrates (CSH), which is responsible for compressive strength in cement-based materials. This, in turn, requires the rice husk to be incinerated at low temperatures (<800°C) to

maximize the amorphous SiO₂ content, which is the most reactive form of SiO₂ to Ca(OH)₂. Most rice husk in Thailand is typically burnt in a boiler at temperature in excess of 1000°C. This causes the amorphous SiO₂, the low-temperature form of SiO₂, to transform to cristobalite, its high-temperature crystalline form, and thus worsens the pozzolanic activity of the resulting RHA. To enhance the use of high-quality RHA as an SCM, a method to characterize its pozzolanic activity must be devised.

Good pozzolanic activity in RHA results from high specific surface area (100-200 m²/g), small particle size (<10 μm), low carbon content (<6-8% by weight), and most importantly, high amorphous SiO₂ content (80-90% by weight), among other factors. A number of methods to assess the pozzolanic activity of different pozzolans (not necessarily RHA) have been proposed in the literature. The most direct and also the most complicated is the accelerated chemical method, where the amount of Ca(OH)₂ consumed in the reaction with a pozzolan is measured, as mentioned in (Sánchez de Rojas, *et al.* 1999). The most widely-used method to measure pozzolanic

activity, although the most time-consuming, is the strength activity index method, where the compressive strengths of cement mortars with and without pozzolan are compared in percentage terms, according to ASTM C311. Some other indirect methods, involving monitoring the change in electrical conductivity of $\text{Ca}(\text{OH})_2$ + pozzolan mixture (the so-called conductometric methods), include the methods of Luxán, *et al.* (1989), Tashiro, *et al.* (1994), and McCarter, *et al.* (1996), among others.

Luxán, *et al.* (1989) proposed characterizing pozzolanic activity of natural rock pozzolans using the conductivity change during the first 2 min when 5 g of pozzolan reacts with 200 ml of saturated $\text{Ca}(\text{OH})_2$ solution at 40°C. The conductivity change during the first 2 min ($\Delta\sigma$), where adsorption phenomena were predominant, permitted a broad classification of materials into three groups, including non-pozzolanicity ($\Delta\sigma < 0.4$), variable pozzolanicity ($0.4 \leq \Delta\sigma \leq 1.2$) and good pozzolanicity ($\Delta\sigma > 1.2$). Instead of using saturated $\text{Ca}(\text{OH})_2$ solution, Tashiro, *et al.* (1994) studied the change in electrical resistivity (the reciprocal of conductivity) when a number of pozzolans were mixed with $\text{Ca}(\text{OH})_2$ powder and water with a consistency of a paste. The rise in resistivity of the pastes measured under steam curing at 70°C correlated well with $\text{Ca}(\text{OH})_2$ consumption as studied by XRD. However, since the measurements were made when the pastes were subjected to elevated-temperature steam curing, the tests gave no information on the reaction rate at ambient temperature.

Using an impedance method, McCarter and Tran (1996) measured the conductivity change of various pozzolans (not including RHA) mixed with $\text{Ca}(\text{OH})_2$ powder and water for about 48 h, using a fixed frequency of 5 kHz. This frequency was believed to be sufficient to reduce the polarization effects at the electrodes to a negligible amount. Using an alternating current source, unlike direct current as in the method of Luxán, *et al.* (1989) four regions in the conductivity drop could be identified and related to different phenomena involving pozzolanic reaction. The most marked drop in conductivity and the time at which it occurred were combined to rank the materials tested based on their pozzolanic activity.

In the present work, the authors adopt the electrical methods of Luxán, *et al.* (1989) (direct

current measurements) and McCarter and Tran (1996) (alternating current measurements) to evaluate the pozzolanic activity of several types of RHA. The applicability of both methods will be discussed in comparison to the strength activity index and other physical and chemical properties known to influence the pozzolanic activity of RHA.

Materials and Experimental Procedures

Six types of RHA (denoted RHA 1 through RHA 6), resulting from burning rice husk from the same source at different burning temperatures and conditions, were investigated. RHA 1, RHA 2, and RHA 3 were burnt under insufficient O_2 ; while RHA 4, RHA 5, and RHA 6 were burnt under excess O_2 (for complete combustion) at 600°C, 750°C, and 900°C, respectively. After incineration, they were all milled to obtain a mean particle size of $\approx 10 \mu\text{m}$, as confirmed by the particle size distribution analysis (Mastersizer S, Malvern Instrument, UK) in Figure 1. The chemical oxide composition of the RHA was studied by X-ray fluorescence spectroscopy (XRF, Phillips, PW 2404, 4 kW, Netherlands) using boric acid as a binder at a ratio of RHA: boric acid = 3: 1.25 by weight. A CHNS/O analyzer (TruSpec CHN, LeCO, UK) was used to measure the carbon content of the RHA, since XRF is incapable of detecting such light atoms as carbon. The phasal composition of the RHA was investigated by X-ray diffraction (XRD) technique (JEOL, JDX 3530, 2kW, Japan). The specific surface area was investigated by nitrogen adsorption technique (Autosorb-1, Quanta Chrome, USA) after outgassing at 300°C for 6 h. The amorphous SiO_2 content was determined from the weight loss of the RHA sample after being washed in a boiling 4 M KOH solution for 3 min, following the Spanish Standards.^(8,9)

Following the electrical method of Luxán, *et al.* (1989), 5 g of each RHA was added to 200 ml saturated $\text{Ca}(\text{OH})_2$ solution at 40°C and the conductivity was measured every 0.5-1 min for 1 h using a portable conductivity meter (PC 300 series, OAKTON, Singapore) with a cell constant, k , of 1.0 cm^{-1} . The measurements were made under constant stirring. The conductivity values compensated at 25°C (using a temperature coefficient of $0.021/^\circ\text{C}$) are reported, to eliminate the effect of temperature on conductivity.

Adopting the method of McCarter and Tran (1996) each RHA was activated by $\text{Ca}(\text{OH})_2$

and water to form a paste, using a fixed RHA: Ca(OH)₂ ratio of 9: 1 and a water/solid ratio of 0.9 by weight. The ratios were used to maintain a castable consistency of the pastes. The pastes were cast into a sealed 25 mm x 25 mm x 25 mm polycarbonate mold, with a pair of stainless steel electrodes (24 mm x 38 mm x 1 mm) attached on two opposite sides. The conductivity measurements were made using an Autolab PGSTAT302 (ECO CHEMIE, Netherlands) at 5 kHz of frequency, under a fixed amplitude of 0.35 V, and no bias. The measurements were made every 15 min from as early as 5 min (from the time when water was added to the solid mixture) to about 52 h under room temperature (25°C) in a 100% relative humidity chamber. The resistance (R) at each time was then converted to conductivity (σ) using the length (L) and the cross-sectional area (A) of the sample according to the relationship: $\sigma = L/(RA)$. The conductivity was normalized by the earliest conductivity (at $t \approx 5$ min) to account for the conductivity contribution from other pre-existing salts in the pastes. Finally, the strength activity indexes of cement mortars containing 10% RHA (by weight of binders) at 3, 7, and 28 days were obtained according to ASTM C311 for blended cements under a fixed flow condition. The reported strength activity index was an average of three replicates for each RHA sample.

Results and Discussion

The physical properties of the RHA are shown in Table 1. As mentioned in the previous section, the mean particle sizes of all RHA were controlled by milling to be approximately the same at 10 μm . Their particle size distributions were also very similar, as shown in Figure 1. In contrast to the previous finding by Nair, *et al.* (2006) that RHA with lower carbon content would exhibit an improved pozzolanic property, the RHA with low carbon contents in the present work (RHA 4, RHA 5, and RHA 6) do not necessarily yield higher compressive strengths than others with higher carbon contents. Moreover, the systematic increase in specific surface area from RHA 6 to RHA 1 does not necessarily lead to a systematic increase in compressive strengths at all ages considered, contradicting the previous finding by (Payá, *et al.* 2001). The high specific surface areas in RHA 1, RHA 2, and RHA 3 might mainly be the result of the high carbon contents, since carbon in the form of ash is known to possess very high specific surface area ($\approx 1000 \text{ m}^2/\text{g}$, according to Nehdi, *et al.* (2003))

due to its microporous structure. It is, therefore, not surprising that the RHA with high carbon content would also exhibit very high specific surface area, although this would cause an adverse effect on the pozzolanic activity.

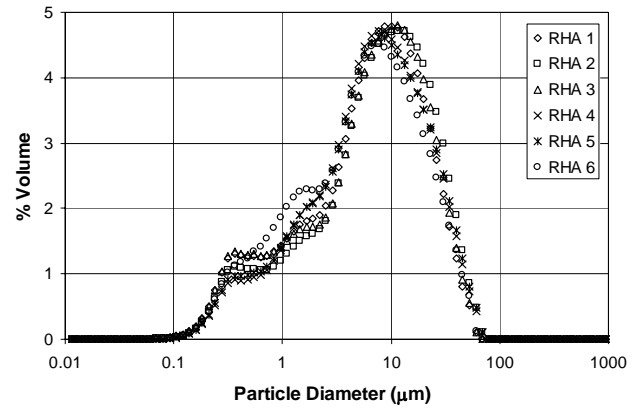


Figure 1. Particle size distributions of the RHA.

Table 1. Physical properties of the RHA

	RHA 1	RHA 2	RHA 3	RHA 4	RHA 5	RHA 6
Specific surface area (m ² /g)	287.13	160.00	126.03	104.50	48.69	9.57
Mean particle size (μm)	9.24	11.01	9.69	9.92	10.05	8.83
Carbon content (wt %)	5.64	5.93	7.56	0.70	0.38	0.47
Amorphous SiO ₂ content (wt %)	83.81	82.27	62.38	90.94	85.38	38.77
Chemical oxide composition (wt %)						
SiO ₂	91.39	93.14	93.59	93.02	92.12	93.22
Al ₂ O ₃	1.22	0.68	0.89	0.69	0.78	0.70
Fe ₂ O ₃	0.34	0.30	0.31	0.28	0.28	0.27
CaO	4.12	2.94	2.28	3.03	3.94	2.81
MgO	0.64	0.61	0.61	0.69	0.69	0.65
SO ₃	0.22	0.22	0.21	0.20	0.19	0.17
Na ₂ O	-	-	-	-	-	0.19
K ₂ O	1.16	1.24	1.30	1.22	1.14	1.16
P ₂ O ₅	0.84	0.80	0.82	0.80	0.78	0.76
MnO	0.08	0.07	-	0.07	0.08	0.07
Strength activity index (%)	(mortar with 10% RHA, fixed flow condition)					
3 days	131	126	116	125	121	115
7 days	125	127	115	122	120	113
28 days	127	131	119	126	129	120

Regarding the chemical oxide composition, the SiO₂ contents for all RHA are very similar, in the range 91-93%. However, these numbers only reflect the total SiO₂ content of the RHA, including all forms of SiO₂, e.g., cristobalite, quartz, and amorphous SiO₂. Considering only the amorphous

SiO₂ content, Table 1 shows that RHA with low amorphous SiO₂ contents (RHA 3 and RHA 6) tend to yield lower compressive strength than the rest at all ages considered, in agreement with the work of (Payá, *et al.* 2001). It should be noted that RHA 3 and RHA 6 exhibit approximately the same compressive strengths at all ages, despite their vast difference in the amorphous SiO₂ contents, and that the highest amorphous SiO₂ content in RHA 4 does not necessarily lead to the highest compressive strength at any of the ages considered. The low amorphous SiO₂ contents in RHA 3 and RHA 6 are confirmed by the presence of cristobalite peaks at $2\theta \approx 22^\circ$ in the XRD pattern of Figure 2. For the rest of the RHA, their XRD patterns only show broad peaks centered about $2\theta \approx 22^\circ$, which are characteristic of amorphous SiO₂. Some amounts of quartz and calcite (CaCO₃) are also detected in Figure 2. The presence of calcite might be explained by the reaction between CaO from the RHA and CO₂ liberated during the incineration, while the presence of quartz might result from some contaminations with sand from rice fields. The above observations point to the fact that the pozzolanic activity of RHA, as partly reflected by the strength activity index method, cannot be assessed by considering any one of these factors separately. It is rather the result of the combined effects of all these physical factors.

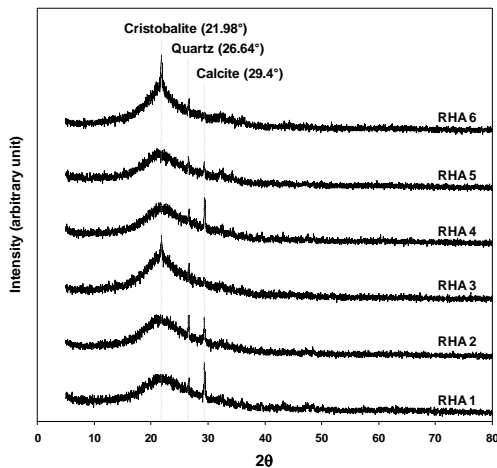


Figure 2. X-ray diffractograms of the RHA.

The conductivity measurements following the method of Luxán, *et al.* (1989) are shown in Figure 3 (a) for the duration of 60 min and (b) for the duration of only 10 min. The dissolved Ca²⁺ and OH⁻ ions in the saturated Ca(OH)₂ solution make it electrically conductive. When the RHA was added, some of these ions were consumed by the pozzolanic reaction with reactive SiO₂ from the

RHA. The conductivity of the mixture is thus reduced as the reaction proceeds over time. This forms the basis for the evaluation of the pozzolanic activity by a conductivity method; the faster the conductivity drops, the higher is the pozzolanic activity. In Figure 3 (a), RHA 1 shows the fastest conductivity drop and the conductivity seems to continue to decrease at a constant rate beyond 60 min. On the other hand, RHA 6 shows no significant change in the conductivity over the whole duration of the test, suggesting the very poor pozzolanic activity of RHA 6. The rates of conductivity change of RHA 2, RHA 3, RHA 4, and RHA 5 are only intermediate between that of RHA 1 and RHA 6. The second column of Table 2 shows the numerical values of the conductivity change during the first 2 min ($\Delta\sigma$ 2 min) following the method of (Luxán, *et al.* 1989). At longer times, the conductivity changes are only slightly larger, as shown in the third and fourth columns of Table 2, for the time of 5 min and 60 min, respectively. Based on the classification criteria of Luxán, *et al.* (1989) (using $\Delta\sigma$ 2 min), RHA 6 falls under the category of non-pozzolanicity ($\Delta\sigma < 0.4$), RHA 3 and RHA 5 under variable pozzolanicity ($0.4 \leq \Delta\sigma \leq 1.2$), and RHA 1, RHA 2, and RHA 4 under good pozzolanicity ($\Delta\sigma > 1.2$).

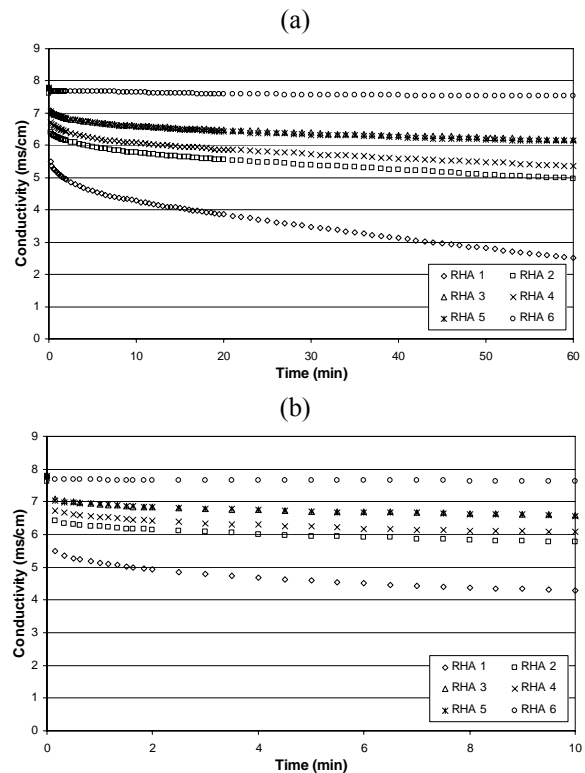


Figure 3. Conductivity measurements following the method of Luxán, *et al.* (1989) over 60 min (a) and 10 min (b).

Table 2. Data summary from both conductivity methods

RHA	$\Delta\sigma$ 2 min	$\Delta\sigma$ 5 min	$\Delta\sigma$ 60 min	$(d\sigma/dt)_{\max}$ (S/m/h)	T (h)	PI (S/m/h ²)	Slope (σ/σ_0) vs t t = 0-24 h (h ⁻¹)
RHA 1	-2.86	-3.20	-5.27	0.041	3.50	117.78	-0.040
RHA 2	-1.48	-1.68	-2.67	0.024	3.50	68.90	-0.036
RHA 3	-0.97	-1.10	-1.62	0.081	19.53	41.61	-0.025
RHA 4	-1.33	-1.51	-2.39	0.046	3.50	130.27	-0.039
RHA 5	-0.96	-1.08	-1.65	0.136	6.65	204.64	-0.039
RHA 6	-0.14	-0.14	-0.27	-0.020	4.02	-49.73	0.014

$$(PI)=(d\sigma/dt)_{\max}/T \times 10^4 \text{ (S/m/h}^2\text{)}$$

Table 3 shows the correlation coefficients between the measured conductivity changes at these three different times and the strength activity indexes (SAIs) at the age of 3, 7, and 28 days. The conductivity change at 2 min shows correlation coefficients of -0.91, -0.77, and -0.49 with the SAIs at 3, 7, and 28 days, respectively. The changes at 5 min and 60 min give approximately the same correlation coefficients, suggesting that taking measurements for longer time does not really improve the efficacy of the method. Moreover, the conductivity changes at these three different times all show decreasing correlation coefficients with the SAIs at later ages. This suggests that the method of Luxán, *et al.* (1989) is only efficient for evaluating the early-age pozzolanic activity of the RHA, or that the compressive strengths at later ages, especially at 28 days, of mortar containing RHA cannot be explained by the pozzolanic activity of the RHA alone. Despite the very high correlation coefficients with the SAIs at early ages, this method only gives a low correlation coefficient of about -0.65 with the amorphous SiO₂ content.

Table 3. Correlation coefficients between the factors derived from electrical measurements and the strength activity indexes (SAIs) and the amorphous SiO₂ content (SiO₂ amorphous)

	3d SAI	7d SAI	28d SAI	SiO ₂ amorphous
$\Delta\sigma$ 2 min	-0.91	-0.77	-0.49	-0.65
$\Delta\sigma$ 5 min	-0.91	-0.77	-0.50	-0.66
$\Delta\sigma$ 60 min	-0.92	-0.77	-0.49	-0.64
PI	0.54	0.54	0.68	0.88
Slope σ/σ_0 24 h	-0.73	-0.77	-0.70	-0.97

McCarter and Tran (1996) identified four regions in the conductivity change when a number of pozzolans were mixed with Ca(OH)₂ powder and water. Region I, characterized by the conductivity drop by about 10% of the initial value, suggested some initial chemical reactivity on the particles. This was followed by a reduction in chemical activity in Region II, known as the dormant period, where a low rate of conductivity change was observed. Region III was where the most marked drop in conductivity occurred. It was believed to correspond to the increased rigidity of the paste via setting. Finally, the reduction in the rate of conductivity drop occurred again in Region IV and was attributed to the decline in chemical activity when the sample became solid. The sharpest conductivity drop in Region III, quantified by the maximum derivative of conductivity, $(d\sigma/dt)_{\max}$, and the time, T, at which it occurred, were used to define the pozzolanicity index or PI.

In the present work, the variations of conductivity and normalized conductivity vs. time are presented in Figure 4 (a) and (b), respectively. For RHA 1, RHA 2, and RHA 4, Regions I and II are absent and only Regions III and IV are detected, similar to the behavior of micro silica as investigated by McCarter and Tran (1996). RHA 3 and RHA 5 show relatively steeper conductivity drops (>10% of the initial value) in Region I, followed by Region III and Region IV, without a dormant period (Region II). This is also similar to the behavior of ground granulated blast furnace slag as observed by (McCarter and Tran 1996). The most surprising behavior is observed in RHA 6, where the conductivity essentially shows an *increase* over time up to about 24 h, after which it starts to decrease until the end of the test period.

The initial increase in conductivity may be due to the increased Ca^{2+} and OH^- ion concentrations after reacting with only a very limited amount of SiO_2 (only ≈ 39 wt%) from RHA 6. The rate at which these ions are dissolved in the solution is faster than the rate at which they are consumed by the pozzolanic reaction. The conductivity drop after 24 h of this RHA is believed to be the result of decreased ion mobility due to drying water, and not from the pozzolanic reaction.

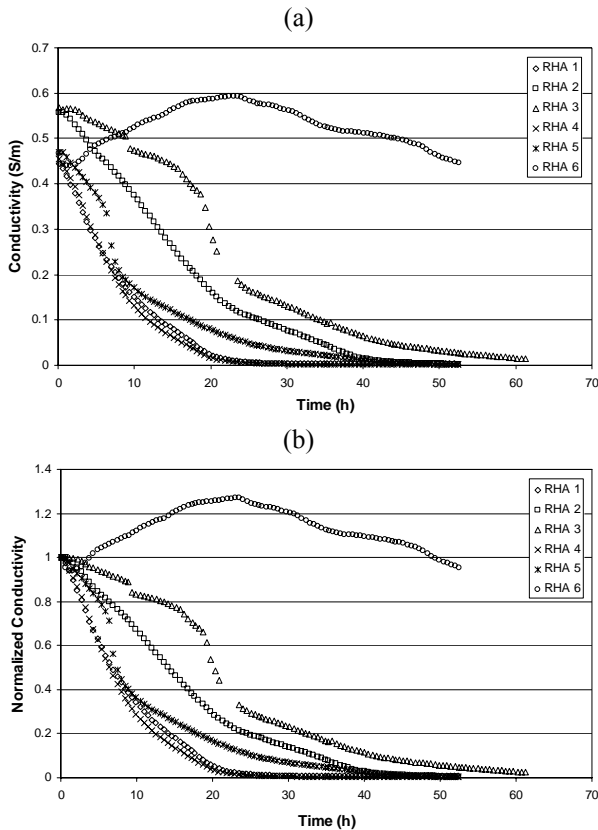


Figure 4. Conductivity (a) and normalized conductivity (b) of $\text{Ca}(\text{OH})_2 + \text{RHA}$ pastes, following the method of (McCarter and Tran 1996).

The variations of the derivative of conductivity change, $(d\sigma/dt)$, with time are shown in Figure 5 and the corresponding parameters for the calculation of PIs are tabulated in Table 2. The high PIs for RHA 1, RHA 4, and especially RHA 5 agree well with the high compressive strengths at 28 days for mortars with these RHA. RHA 6 displays a negative PI, as expected from the increased conductivity in Figure 4 and its low amorphous SiO_2 content. However, the PI for RHA 2 seems to be too low to explain its very high compressive strength at 28 days. From Table 3, the correlation coefficients between the PIs and the SAIs at 3, 7, and 28 days are 0.54, 0.54, and 0.68, respectively.

In contrast to the method of Luxán, *et al.* (1989), these correlation coefficients are increasing with the SAIs at later ages. Despite the low correlation coefficients with the SAIs at 3 and 7 days, the PI does give a much improved correlation coefficient of 0.88 with the amorphous SiO_2 content than those from the method of (Luxán, *et al.* 1989). Comparing the correlation coefficients from the methods of Luxán, *et al.* (1989) and McCarter and Tran (1996) it seems that the former might be more accurate for assessing the pozzolanic activity at early age, while the later is more appropriate for later-age pozzolanic activity.

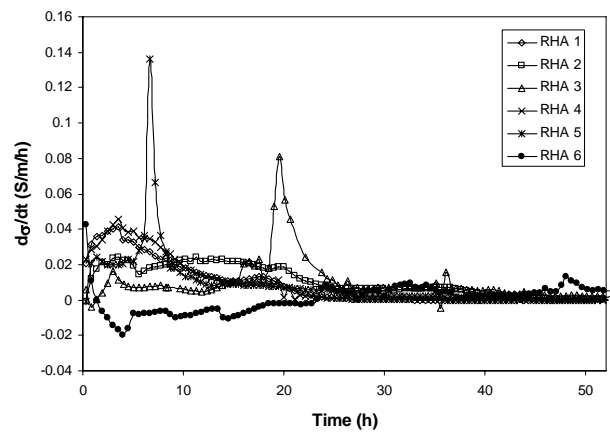


Figure 5. Derivative of conductivity change ($d\sigma/dt$) of $\text{Ca}(\text{OH})_2 + \text{RHA}$ pastes following the method of (McCarter and Tran 1996).

In studying the time-variation of the normalized conductivity (σ/σ_0) in Figure 4 (b), the authors have found that the slope of (σ/σ_0) vs. time during the first 24 h (where the effect of drying water is not yet pronounced) gives reasonable correlation coefficients of -0.73, -0.77, and -0.70 with the SAIs at 3, 7, and 28 days, respectively. To the compressive strength at 28 days, this number (0.70) is only comparable to that derived from PI (0.68), but to the early-age compressive strengths, this method proves to be a better alternative. Compared to the method of Luxán, *et al.* (1989), this method still gives a lower correlation coefficient with the SAI at 3 days. Nevertheless, this method seems to be less time-sensitive than the $\Delta\sigma$ method of Luxán, *et al.* (1989) and the PI method of McCarter and Tran (1996) as the correlation coefficients stay unchanged with the SAIs at later ages. Furthermore, it gives an extremely good correlation coefficient of -0.97 with the amorphous SiO_2 content.

Conclusions

The change in electrical conductivity, as studied by the methods of Luxán, *et al.* (1989) (direct current measurements) and McCarter and Tran (1996) (alternating current measurements), can be used to assess the pozzolanic activity of RHA. However, the two methods are sensitive to pozzolanic activity of the RHA at different ages. The $\Delta\sigma$ method of Luxán, *et al.* (1989) proves to be more accurate in evaluating the pozzolanic activity at early ages, while the PI method of McCarter and Tran (1996) is more appropriate for studying later-age pozzolanic activity. The PI method shows a higher correlation coefficient with the amorphous SiO₂ content than the $\Delta\sigma$ method. Using the slope of the normalized conductivity vs. time during the first 24 h, a much improved correlation coefficient with the amorphous SiO₂ content is observed. This method also yields reasonable correlation coefficients with the strength activity indexes at all three ages considered.

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