A LIMITED PERFORMANCE EVALUATION OF NATURAL POZZOLANS USING THE BULK RESISTIVITY TEST

Final Report
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Introduction

The National Ready Mixed Concrete Association's (NRMCA) research laboratory undertook a limited performance evaluation of five materials that could represent natural pozzolans in mortar mixtures. The evaluation involved the determination of water requirement for target flow, and measurement of compressive strength, and bulk resistivity in comparison to control mixtures for a potential measure of reactivity. Mortar mixtures were prepared with coal ashes that conformed to Class F and Class C in ASTM C618 and slag cement that conformed to ASTM C989. Additionally, one mortar mixture used limestone filler to represent an inert material. The evaluation of strength and resistivity was intended to help distinguish reactive Supplementary Cementitious Materials (SCMs) from inert fillers.

Materials

1. ASTM C150 Type I/II cement meeting ASTM C311-22 Sec 5.3
2. Natural Pozzolans
    a. Calcined clay
    b. Obsidian
    c. Obsidian+Fly ash that did not meet ASTM C618 (referred to as Obsidian+)
    d. Pumice
    e. Rhyolite
3. Class C Fly Ash
4. Class F Fly Ash (low lime)
5. Slag Cement
6. Inert Filler (limestone fines)

Table 1. Mixture Proportions and Flow

Mix ID	**Cement**	**SCM**	**Water**	***w/cm***	**Flow**
ASTM C150 Type I/II cement	740	0	359	0.485	106
Calcined clay	592	148	389	0.526	102
Obsidian	592	148	359	0.485	109
Obsidian+	592	148	379	0.512	102
Pumice	592	148	374	0.505	107
Rhyolite	592	148	359	0.485	107
Class C Fly Ash	592	148	342	0.462	111
Class F Fly Ash (low lime)	592	148	342	0.462	108
Slag Cement	370	370	342	0.462	107
Filler (limestone fines)	592	148	342	0.462	109

A total of 10 mortar mixtures were prepared. Each mixture contained 740 g of cementitious materials and 2035 g of natural silica sand conforming to the requirements for graded natural sand in Specification ASTM C778. The control mixture contained portland cement as the cementitious material at a *w/cm* of 0.485. Slag cement was used at 50% by weight of cementitious mixtures. The mixtures with natural pozzolans, fly ash, or fillers contained these materials at 20% by weight of cementitious material, which is consistent with the proportions required for the strength activity index in ASTM C311. For the test mixtures, the mixing water content was adjusted to attain a mortar flow within 5% of the control mixture as required in ASTM C311.

Curing and Testing

Mortars were mixed in accordance with ASTM C305 and flow was measured in accordance with ASTM C1437. For each batch, nine 2-in. mortar cubes were cast in accordance with ASTM C109. The cubes were cured for 1 day in the mold in a moist room with their upper surfaces exposed to the moist air but protected from dripping water. After demolding, the cubes were immersed in lime-saturated water at 73.5±3.5 °F. Fig. 1a-d shows some of these details.

At an age of 7 days, the bulk resistivity of two cubes was measured in accordance with a modified version of ASTM C1876 (Fig. 2). ASTM C1876 measures resistivity on 100 mm by 200 mm concrete cylindrical specimens cured in lime-saturated simulated pore solution. This study measured resistivity on 2-in. mortar cube specimens cured in lime water. Following the resistivity test the cubes were tested in compression.

At this age, three cubes were immersed in lime-saturated water at 100.4±3.6 °F. This curing option is consistent with the accelerated moist curing defined ASTM C1202. The other 3 cubes were cured in lime-saturated water at 73.5±3.5 °F.

After 28 days, all the cubes were removed from the lime water and the bulk resistivity was measured followed by compressive strength testing.

It is important to recognize that the mortar cubes with different SCMs had different *w/cm* which will impact the hardened properties.

Test Results

The measured compressive strength and bulk resistivity are reported in Table 2. The measured properties of the mixtures containing SCMs or filler were divided by the measured properties of the control portland cement mixture to calculate index values. The following values are plotted and discussed for each batch:

1. Water requirement, %
2. 7-day strength activity index (7-day SAI) and bulk resistivity index (7-day BRI), %
3. 28-day strength activity index (28-day SAI) and bulk resistivity index (28-day BRI), %
4. 28-day accelerated curing strength activity index (28-day SAI A) and bulk resistivity index (28-day BRI A), %

A water requirement of less than 100% shows lower water demand for target flow compared to the control mixture. An SAI or BRI less than 100% indicates that the SCM has a lower strength or resistivity than the portland cement control at that age.

Table 2. Measured Compressive Strength and Bulk Resistivity of Mortars

Mix ID	Compressive Strength, psi			Bulk Resistivity, Ω-m		
	7-day	28-day	28-day accelerated	7-day	28-day	28-day accelerated
ASTM C150 Type I/II cement	3760	4590	4690	31.3	36.2	45.2
Calcined clay	3890	5770	5720	41.2	102.2	153.2
Obsidian	3820	5340	6780	31.4	100.8	494.1
Obsidian+	3490	5270	6110	23.6	48.5	186.1
Pumice	2520	3870	4810	22.6	38.3	145.1
Rhyolite	3660	4380	5070	28.4	56	245.2
Class C Fly Ash	3330	4300	6550	22.8	39.7	125.3
Class F Fly Ash (low lime)	2340	3150	5490	26.4	103.4	737.6
Slag Cement	4340	6810	6040	50.1	109	192.2
Filler (limestone fines)	1540	2310	2250	25.2	35	38.7

Water Requirement

ASTM C618 requires a maximum water requirement of 105% for all fly ashes and 115% for natural pozzolans. The water content and *w/cm* of the mortar mixtures at a similar flow to control are listed in Table 1. Fig. 3 is a plot of the water requirement as a percent of the control. Limestone filler, slag cement, Class F fly ash, and Class C fly ash show a water reduction. Mixtures containing pumice, obsidian+, and calcined clay had an increased water requirement compared to the control mixture. Obsidian+ and Calcined clay had a water requirement exceeding 105% but still below 115%. Mixtures with obsidian and rhyolite had similar water demand to that of the control mixture.

Strength Activity Index (SAI)

In accordance with ASTM C618, the SAI for coal ashes and natural pozzolans at 7 and 28-day age should not be less than 75%. Fig. 4 is a plot of the SAI of the mixtures at these ages and includes the accelerated curing condition. In general, the 28-day SAI A was higher than the 28-day SAI which was higher than the 7-day SAI. The accelerated curing condition promotes a faster pozzolanic reaction compared to the standard curing condition at 28 days. The 7 and 28-day SAI for the Class F fly ash and 7-day SAI for the pumice did not meet the ASTM C618 minimum limit. But note that the ASTM SAI limits are applicable for composite samples of the SCM which is formed by combining equal portions of multiple samples taken from the material that the end user receives. Accelerated temperature curing appears to increase the fly ash strengths more than they do for other SCMs. The SCMs that had the highest water requirement limit (Obsidian+ and Calcined clay) had some of the highest SAI values. Some have argued that these SCMs have a higher porosity and absorb water much like highly absorptive aggregates, and therefore the absorbed water should not be included as part of the mixing water. The higher absorption values of these SCMs need to be established though. The considerably low SAI of the mortar mixture with limestone filler cannot be explained and will likely need to be retested.

Bulk Resistivity Index (BRI)

Fig. 5 is a plot of the BRI. In general, the 28-day BRI A was higher than the 28-day BRI which was higher than the 7-day BRI for the different curing conditions. Unlike the SAI, BRI values appear to be more sensitive to the impact of the SCMs. The mortar containing filler had a BRI of around 100% suggesting no change when compared to the portland cement control. The lowest 28-day BRI A among all SCMs was 277% for the Class C fly ash. The 28-day BRI A for the Class F fly ash and Obsidian mortars were 16 times and 11 times greater than that of the portland cement mortar.

Best Metric to Differentiate Reactive SCMs from Portland Cement and Fillers

Fig. 6 is a plot of the 7-day SAI and 7-day BRI. Mixtures with SCMs underperformed compared to the portland cement control mixture at 7 days. At this age, neither SAI nor the BRI can effectively classify reactive SCMs from fillers.

Fig. 7 is a plot of the 28-day SAI and 28-day BRI. By 28 days, the SCMs have started showing their effectiveness and BRI provides a better indicator of reactivity than SAI. The BRI of the mixture with filler was 97%; whereas the BRI for the least reactive SCMs - pumice and Class C fly ash was 106% and 110%, respectively. This is not a significant difference to suggest a higher degree of reactivity of these SCMs.

Fig. 8 is a plot of the 28-day SAI A and 28-day BRI A. The 28-day BRI A is substantially higher than the 28-day SAI A. This is consistent with concrete performance where SCMs may have a small impact on compressive strength but a substantial impact on bulk resistivity particularly at later ages. These data suggest that 28-day BRI A is the best option to distinguish the reactivity of SCMs from inert fillers. It also indicates an improved performance for the potential durability of mixtures with these SCMs compared to the mixture with only portland cement. Based on this limited scope of work, it is suggested that a 28-day BRI A of 200% (accelerated curing) is a good metric to differentiate reactive SCMs from fillers.

Bulk Resistivity Test

Bulk resistivity is an indicator of a material's resistance to the penetration of fluids and dissolved aggressive ions and is related to diffusivity through the formation factor. The formation factor of a porous body is a material property and is an inverse function of the porosity (ϕ) and pore connectivity (β). The formation factor is also related to concrete resistivity, RCP, and diffusivity and can be calculated as (Weiss et al. 2017; Obla 2019):

$$F \cong \frac{1}{\phi\beta} = \frac{\rho}{\rho_0} = \frac{D_o}{D_{eff}} = \frac{\sigma_0}{\sigma} = \frac{(180{,}828 \times \sigma_0)}{Q} \qquad (1)$$

Where:

ρ = resistivity of the concrete,
ρ_0 = resistivity of the pore solution in the concrete,
σ = conductivity of the concrete,
σ_0 = conductivity of the pore solution in the concrete,
Q = RCP value of the concrete in coulombs measured in accordance with ASTM C1202 on specimens with a diameter of 3.74 in. (95 mm) and length of 2 in. (50.8 mm),
D_{eff} = Effective chloride ion diffusion coefficient, and
D_o is the self-diffusion coefficient of chloride ions ($2.03 \text{ X}10^{-9}$ m^2/s at 25 °C).

The bulk resistivity test has several advantages:

1. It is a rapid and repeatable test that is easy to perform and does not involve extensive specimen preparation (such as that required for RCP, ASTM C1202)
2. It is a non-destructive test and the same specimens can be tested for compressive strength
3. The accelerated conditioning used in this project requires a 100.4±3.6 °F room which is typically found in many commercial laboratories that perform ASR testing (ASTM C1293)
4. Unlike strength, resistivity is useful in differentiating the performance of SCMs from inert fillers and the cement-only control mixture.

The lime-saturated simulated pore solution is the stated conditioning method in ASTM C1876. The reason is related to the leaching of ions from the test specimens having an impact on the measured resistivity – generally increasing the measured value. A modification in the ASTM C1876 BR test employed in this study is subjecting the specimens to lime-water curing as opposed to simulated pore solution curing in ASTM C1876. A past study (Obla and Lobo, 2021) showed that for specimens immersed in simulated pore solution, the BR measurements continued to drift upwards and did not reach a stable value with time. The results did not drift for specimens subject to lime water curing. Lime water immersion is easier and more practical for technicians, laboratories, and testing agencies as they have more experience with it. It is safer to handle, less expensive, and easier to dispose of the lime water solution compared to the simulated pore solution. Another issue is that test specimens for strength tests are cured in a moist room or immersed in lime-saturated water and therefore the same specimens can be tested in BR before strength testing.

As shown in Eqn. 1 there is a direct relationship between BR and RCP as long as the curing conditions are similar. Practitioners familiar with RCP criteria for classifying the chloride penetrability of concrete mixtures can calculate the corresponding BR criteria. BR of specimens subject to lime water curing has been correlated with ASTM C1202 (Obla and Lobo, 2021) which has been correlated with the ASTM C1556, chloride diffusion coefficient (Obla et al. 2016). A different set of BR criteria may have to be developed for specimens subject to lime-saturated simulated pore solution curing.

Possible Approach to Using the Bulk Resistivity Test in ASTM Standards

The ASTM C09.24 Subcommittee on Supplementary Cementitious Materials (SCMs) is developing a performance specification for Supplementary Cementitious Materials. However, unlike the ASTM C1157 performance specification for cement which has performance requirements such as strength, sulfate resistance, high early-strength, alkali-silica resistance, and heat of hydration the proposed SCM standard has only a strength activity index and an SCM reactivity index as performance requirements. The standard does not identify SCMs that can meet other performance requirements. One of the main reasons for incorporating SCMs is to reduce penetrability. Incorporating a required 28-day BR A value, such as a minimum of 90 Ω-m (which corresponds to an ASTM C1202 value of 2000 coulombs) will ensure that the SCM is capable of producing concrete with low penetrability. Fig. 9 is a plot of the 28-day BR A and 28-day BRI A.

The subcommittee is also developing a specification for natural pozzolans and looking at relaxing some of the requirements for coal ash in ASTM C618 to increase marketable material. Incorporating a required min. BRI A limit of 200% can help differentiate reactive SCMs from fillers.

As discussed before, the accelerated moist curing performance is in accordance with ASTM C1202. Accelerated moist curing is not a new concept for SCM specifications. The silica fume specification, ASTM C1240, requires an accelerated pozzolanic strength activity index where the specimens after 24 h of initial curing in the moist room (23 ± 2 °C and relative humidity of not less than 95 %) are placed in airtight glass containers and stored at 65 ± 2 °C for six days.

A Final Comment on the Need for Performance-Based Specifications in Standards

ACI 318-19 specifically states maximum *w/cm* in its durability provisions for concrete members. The *w/cm* requirements vary between 0.40 and 0.55. In this study, the *w/cm* for the 10 batches varied between 0.46 and 0.53. Fig. 10a and 10b show the 28-day BRI and 28-day SAI plotted against the *w/cm*. It is clear that the SCM type used had a more significant influence on penetrability than *w/cm*. Even though this work was performed on mortar a similar observation is expected for concrete mixtures. This suggests that as opposed to a *w/cm* requirement a performance alternative such as resistivity may be able to categorize mixtures based on their resistance to penetrability. This will make it easier to use SCMs with a higher mixing water requirement.

Acknowledgments

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(a) (b) (c) (d)

Fig. 1. (a) & (b) Cubes being prepared (c) Flow test (d) Cubes immersed in lime water

Fig. 2. Cubes tested in bulk resistivity

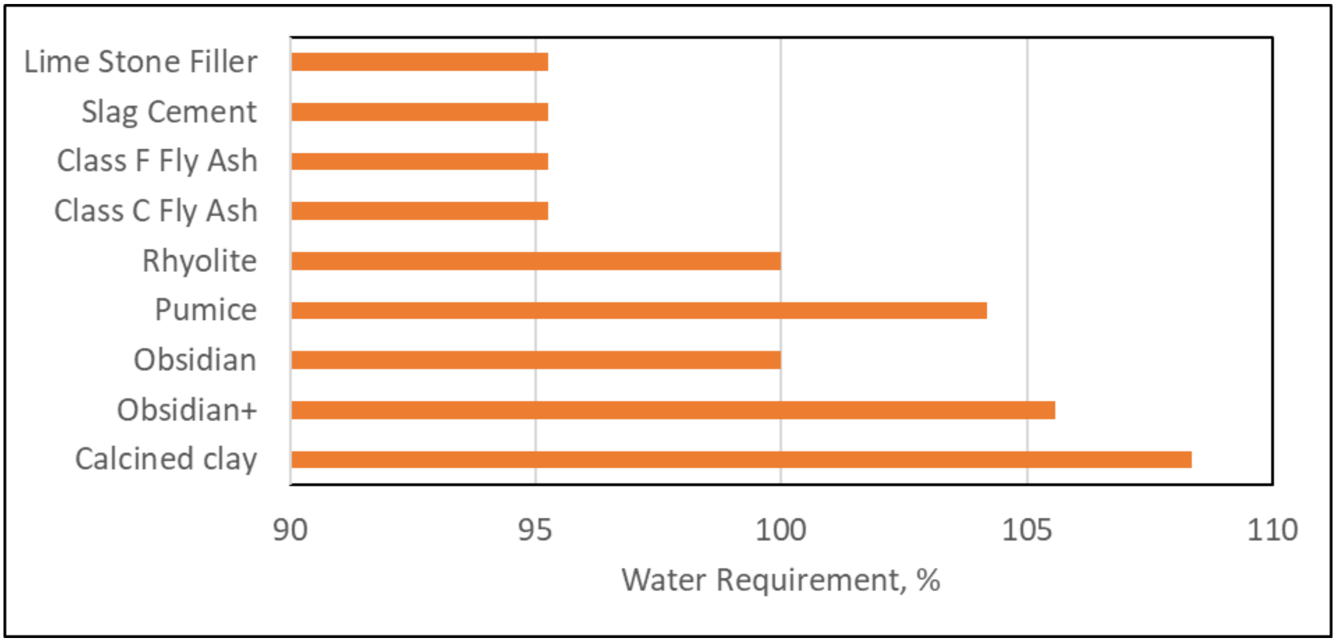

Fig. 3. Water requirement as a percent of the control for different SCMs

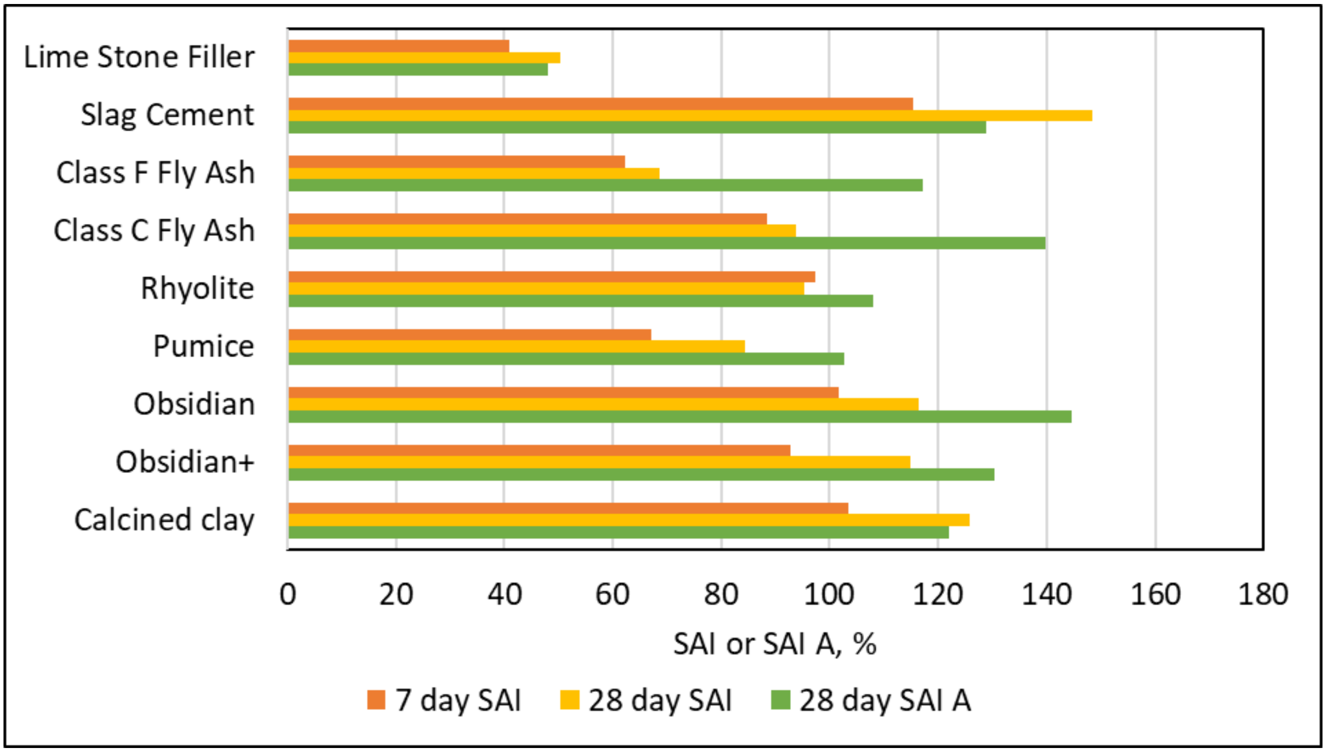

Fig. 4. Strength Activity Index for different SCMs

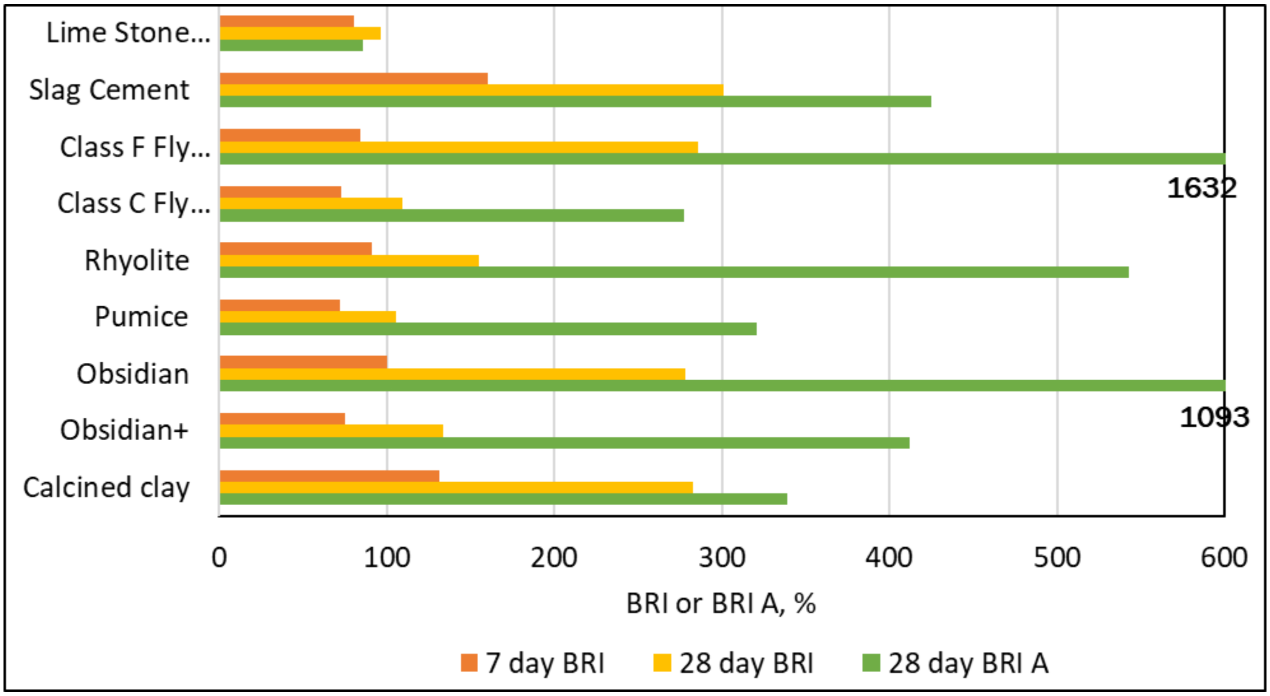

Fig. 5. Bulk Resistivity Index for different SCMs

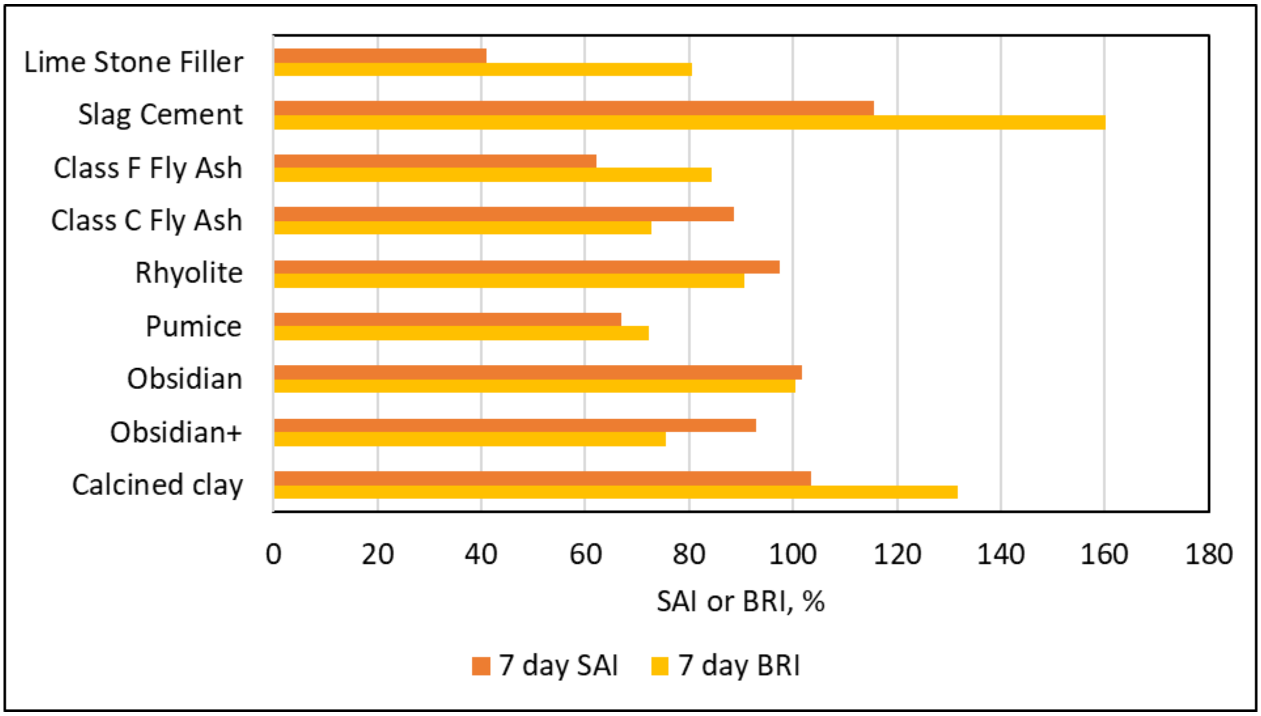

Fig. 6. 7-day Strength Activity and Bulk Resistivity Index for different SCMs

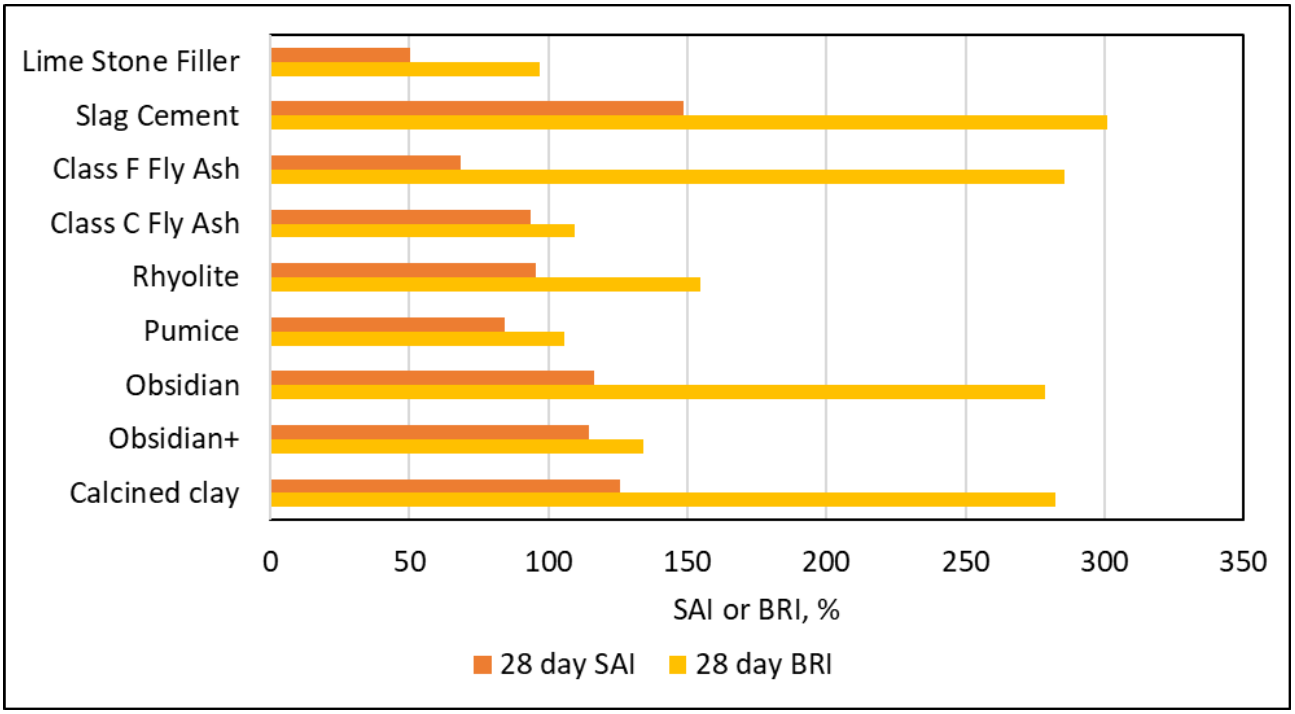

Fig. 7. 28-day Strength Activity and Bulk Resistivity Index for different SCMs

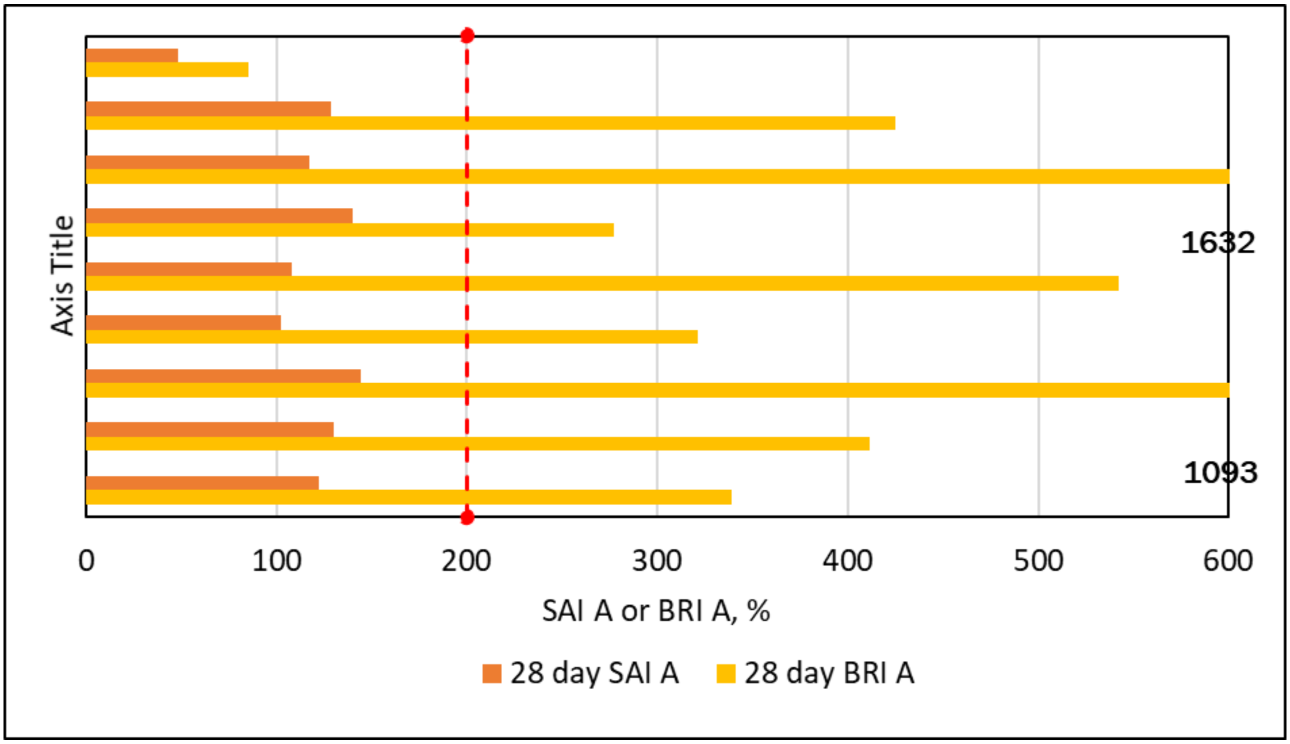

Fig. 8. 28-day Accelerated Strength Activity and Bulk Resistivity Index for different SCMs

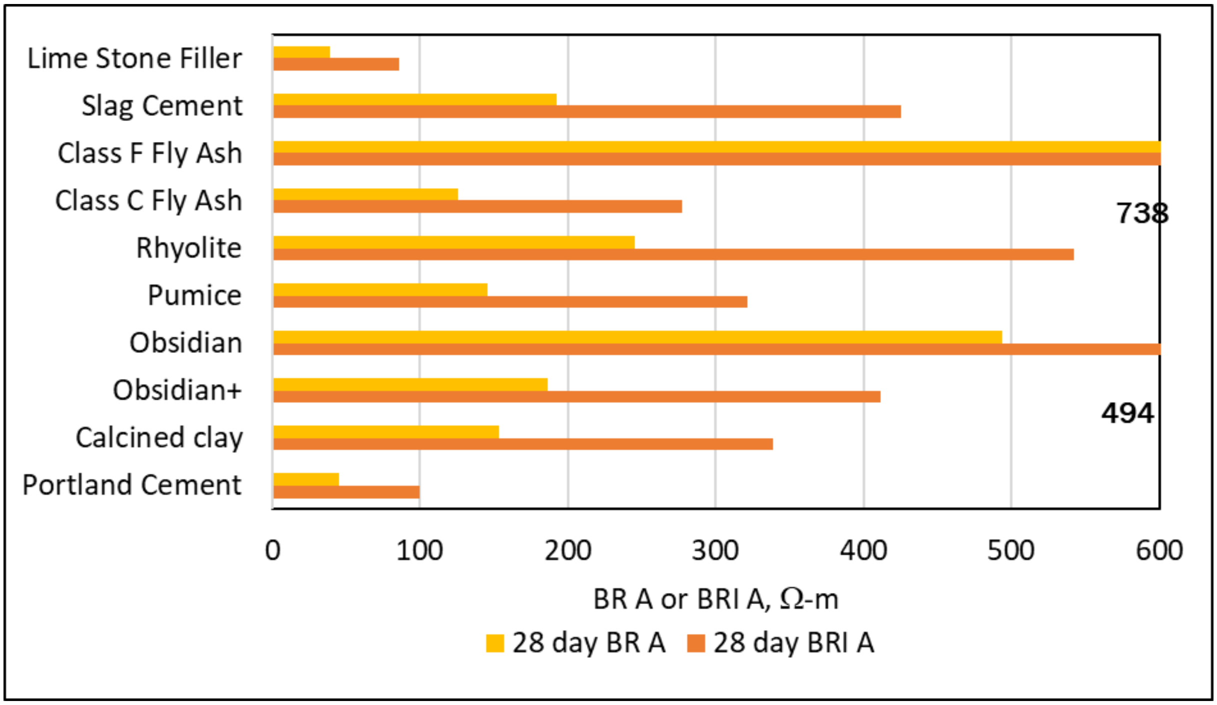

Fig. 9. Impact of Accelerated Conditioning on Bulk Resistivity of different SCMs

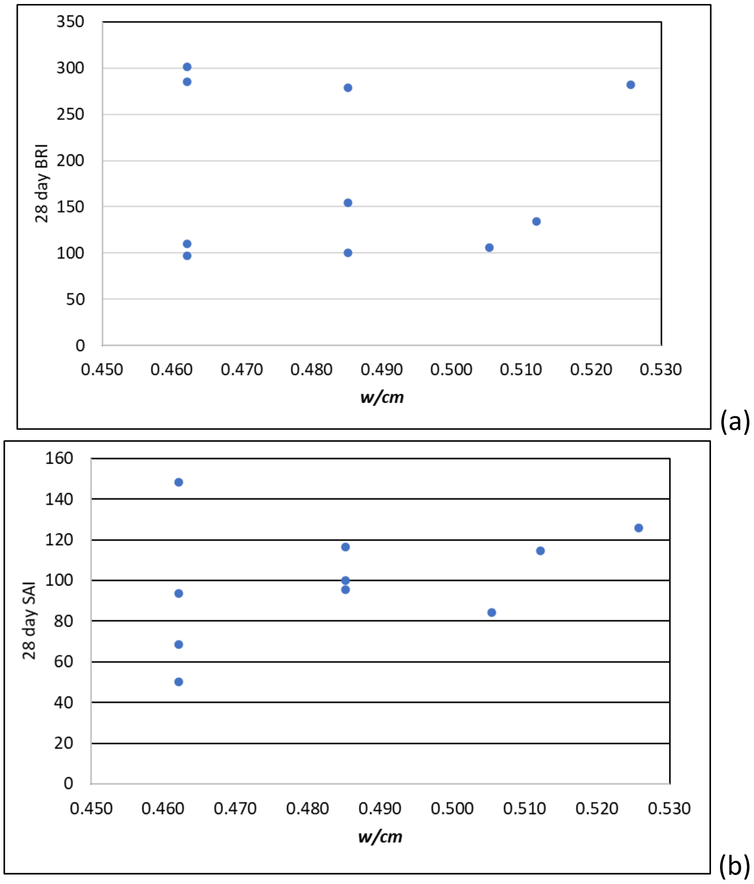

Fig. 10. Effect of *w/cm* on SAI and BRI of all batches Tested in Study