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# SUITABILITY OF ALKALI ACTIVATED BLENDED GGBS/FLY ASH CONCRETE FOR CHLORIDE ENVIRONMENTS

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# ABSTRACT

This study investigates chloride ingress into alkali-activated slag/fly ash (AA-S/F) concretes with various ratios of slag to fly ash (80/20, 60/40, 40/60 and 20/80). Alkaline activators were a mixture of sodium hydroxide and sodium silicate to obtain between 6-8% sodium hydroxide (NaOH) concentration per weight of cementitious material. The $SiO_2/Na_2O$ ratios (silica modulus, Ms) of mixes were 0.45, 1.0 and 1.25. Tests applied included typical fresh and hardened properties, air content, slump, compressive strength, bulk resistivity and non-steady state chloride migration. The results show that while attractive engineering properties can be achieved, the chloride ingress in the AA-S/F concretes studied here with $\geq$ 20% fly ash is moderate to high and this may make them unsuitable for use in reinforced concrete in chloride environments.

**Keywords:** Alkali activated slag/fly ash concretes; workability; compressive strength; bulk resistivity; chloride migration coefficient

## INTRODUCTION

Alkali activated materials (AAMs) have been under consideration as an alternative binder system since at least 1908 [1]. The main limitations facing the commercial adoption of these binders are the challenges in controlling engineering properties when using intrinsically variable waste-derived precursors, and the lack of field performance data, especially in extreme environments. Slag and fly ash are the major by-products used as precursors for making these types of binders. Previous investigations show that alkali-activated high calcium systems such as AAS concretes usually set rapidly, can have low workability, and sometimes also a high degree of drying shrinkage compared to Portland cement (PC) based systems, and all of these are known to affect durability properties [2-6]. Low calcium alkali activated systems, on the other hand, can exhibit high workability and a lower shrinkage than PC systems [7], but need elevated temperatures for curing and hardening. Chi and Huang [8] studied the behaviour of the binder products and properties of alkali-activated slag/fly ash blended (AA-S/F) mortars and concluded that better properties, compared to PC, was obtained in terms of compressive strength, flexural strength and water absorption. Abdalqader et al. found that the highest strength of AA-S/F obtained was attributed to the paste mix consisting of slag: fly ash in a 3:1 ratio. Gao et al. reported that samples with a higher slag/fly ash ratio exhibit a lower level of porosity [9-10]. In this investigation, blended binder systems were used as they provide control of properties such as setting time, workability, shrinkage, strength and durability compared to those based on 100% fly ash or slag. An experimental programme was developed to study the workability, strength development and resistance to chloride ingress for a range of AA-S/F concrete mixes that do not require high temperature curing. Fresh properties, compressive strength at different ages, bulk resistivity and non-steady state chloride migration were determined and reported for various AA-S/F concretes.

## RESEARCH SIGNIFICANCE

100% fly ash based alkali-activated systems often require elevated temperature curing, while slag alone can produce flash setting. Engineering properties such as shrinkage influence the cracking probability and durability properties, and must be better controlled. Therefore, in this programme, the authors are using a range of fly ash/slag blends to avoid elevated temperature curing and offer control over engineering properties.

# EXPERIMENTAL PROCEDURE

## Materials

The primary raw materials used in this study were granulated blast furnace slag (GGBS) and fly ash (named in the UK as pulverised fuel ash, PFA), which were provided by ECOCEM – Ireland and Power Minerals Ltd, UK, respectively. The chemical and physical properties are presented in Tables 1 and 2. GGBS and PFA were blended in 80/20, 60/40, 40/60 and 20/80 mass proportions, to be activated and used as binder.

Sodium hydroxide (NaOH) powders or pellets were dissolved in water to produce the alkaline solutions. The chemical composition of the sodium silicate solution was 15.5% sodium oxide ($Na_2O$), 30.5% silicon oxide ($SiO_2$) and 54% water. In this study, the percentage NaOH in the paste (NaOH%) was selected to be 6 and 8% by powder (GGBS and PFA) weight, and the silicate modulus (molar ratio $SiO_2/Na_2O$) was selected to be 0.45, 1.0 and 1.25, by blending sodium silicate and sodium hydroxide.

The aggregates used in this study were crushed basalts from local sources in Northern Ireland, and comprised 16.5 mm and 10 mm crushed coarse and fine aggregates, and 4 mm sand. These were combined in a ratio of 48:12:40 to get the maximum packing density in (AA-S/F) concrete mixes. Properties of the aggregates are reported in Table 3. Potable tap water (i.e. drinking quality water) was used to make the concrete mixes.

## Mixing

Blended cements was prepared by dry-mixing GGBS and PFA in different proportion ratios with a benchtop food mixer to get a homogeneous blended powder. Then the concrete mixtures were mixed in a laboratory pan-mixer. Crushed basalt aggregates and sand were dry mixed together for a minute and after adding the blended GGBS and PFA powder, mixing continued for 2 minutes. The sodium hydroxide solution was then added, and after 2 minutes of further mixing, sodium silicate solution was added and mixing continued for a further minute. The details of the different mixtures and their essential properties are presented in Table 4.

## Measuring fresh properties

The slump test and flow test was carried out for each mix, and the air content of mixes was measured in accordance with BS EN 12350 [11-12].

## Casting and curing of the specimens

From each concrete mix, nine 100 mm cubes, and three 100x200mm cylinders, were cast for the determination of compressive strength [13], bulk resistivity and chloride migration [14]. The concrete specimens were cast in three layers and compacted on a vibrating table. After casting, all the moulded specimens were covered with plastic sheets and left in the casting room for 24 h. They were then demoulded and kept in a sealed plastic zip bag until the test date.

## Test preparation and procedures

To determine chloride migration in AA-S/F concretes, three slices with a thickness of 50 mm were cut off from each cylinder at an age of 90 days, after removing 5 mm from the top finished surface. A vacuum saturation regime was used to precondition the slices so that the chloride flow is predominantly diffusive and initial sorption or capillary forces do not dominate. The vacuum was applied to remove air for three hours and released afterwards. Samples were wrapped in hessian saturated in deionised water to prevent leaching of ions from them and placed in the container. The weight of the sample was noted after an hour for weight ($W_1$) and then vacuum was applied, followed by further saturation. Weight was checked again ($W_2$) and this was repeated until constant mass was reached. Usually after 6 hours, when $W_i$-$W_{i-1}$ was less than 0.1%, i.e., <1g of change for a 1 kg sample, the samples were considered fully saturated. After conditioning to a surface-dry condition, an epoxy resin (Sika Gard-680S) was applied onto the surfaces of the specimens in three layers except for the two test surfaces (cut surfaces). Chloride penetration depth and non-steady state migration coefficient of these concretes were determined by carrying out the test as outlined by NT BUILD 492 [14].

Bulk resistivity measurement was performed on the same samples before and after the NT BUILD 492 test. The test set-up for measuring resistivity is shown in Figure 2.

# EXPERIMENTAL RESULTS AND DISCUSSION

## Fresh properties and compressive strength

All the mixes had a slump greater than 170 mm. The mix designs for the AA-S/F concretes consisted of using various ratios of slag to fly ash, percentage of alkali and the $SiO_2/Na_2O$ ratio (silica modulus, Ms) to get different grades of AA-S/F concretes as outlined in Table 4.

For all of the mixes, the W/B (given in Table 4) included the total water in the mix including the water from water glass. For increasing the proportion of the fly ash, the W/B for mixes was reduced while the paste content of the mixes was kept constant. All mixes met the minimum slump and compressive strength requirement for their use in chloride environments, such as S2 and C20/25 specified in BS8500-1:2015 for XS (marine) environments. The mix design for all mixes was based on a particle packing optimisation and therefore, the paste is filling in the voids and strength might have been optimised by the aggregate fraction and paste strength.

The slump and flow results of the concretes are presented in Figures 3 and 4. It can be observed that although W/B was adjusted based on the proportion of the fly ash in the system, increasing the fly ash in the blend increased the workability, and the maximum workability can be achieved corresponding to maximum fly ash content in the system. The literature shows that increasing the sodium oxide content results in increased workability, reduced setting times and higher compressive strength. However, high concentrations resulted in loose structure in sodium hydroxide activated samples [16-19]. For mixes with the same slag to fly ash and water to binder ratio, comparing mixes 1, 5, 9 and 13 and mixes 4, 8, 12 and 16 respectively, the slump values of the AA-S/F concretes with the same Ms ratio increased with an increase of NaOH%. The slump values also increased with the increase of Ms which can be seen by comparing odd and even mixes (e.g. mix no. 1 to 2, 3 to 4) in the two groups with two different NaOH% and the same ratios of slag to fly ash.

The air contents of the fresh mixes AA-S/F are presented in Figure 5. The lowest air content was measured for mixes with a slag/fly ash ratio of 60/40 while the highest was for 20/80 (except mix 15). In the mixes made with a higher alkali percentage, the air content is lower (comparing mix no. 1, 5 and 13 to mix no. 4, 8 and 16, respectively) and using higher silica content has had no major effect, especially for mixes with higher alkaline content (comparing mix no. 3 to 4 or 7 to 8). However higher silica content in the activator will be more effective when the fly ash proportion increases in the blend, and this causes higher air content when using higher silica content (comparing mix no. 15 to 16).

Figure 6 shows the compressive strengths of AA-S/F concrete mixes at 2, 28 and 90 days. As is expected, the more fly ash in the system, the lower is the early age and long term strength. An increase in NaOH% and Ms generally increases the compressive strength of AA-S/F concretes, which agrees with the results reported by others. This is due to more N-A-S-H (sodium aluminosilicate hydrate) reaction product being generated, based on the increase of NaOH%, and more C-A-S-H (calcium aluminosilicate hydrate) reaction product being produced, based on the increase of $SiO_2$ content. From the 28 day compressive strengths (Table 4) except mixes no. 7, 11 and 13 to 16, all achieved the required strength for the exposure classes XS3 and XD3. Mixes no. 7, 11, 13, 14 and 16 seem to meet the strength requirements for the exposure classes XS1, XD1 and XD2.

## Resistivity and Diffusivity

Figure 7 depicts the average bulk electrical resistivity for different AA-S/F concrete mixtures, respectively. It shows the bulk resistivity before and after testing for the specimens used in the migration test (NT BUILD 492). A higher resistivity could be attributed to a denser structure with low connected porosity and/or less conductivity of the binder matrix, and such mixes will be expected to be superior in resisting ionic (chloride) flow. Higher slag content in AA-S/F concretes resulted in higher bulk resistivity, while mixes with higher alkali content show higher bulk resistivity when slag dominates the blend. Furthermore, bulk electrical resistivity decreases for mixes made with higher silica modulus (except mixes 3 and 4), whereas the bulk resistivity seems to be a maximum at NaOH=8% and Ms=1.0 when S/F=80/20. This may be related to the alkali content which is required to produce maximum reaction products.

The results show that the resistivities before and after the NT BUILD 492 test are relatively close for half of the mixes, with the exception of mixes no. 1, 3, 4, 7, 8, 9, 11 and 15, where the trend is for a reduction in resistivity after testing. The change in resistivity can be related to the degree of damage the structure is undertaking as part of the migration test, and/or changes in pore solution chemistry, maybe due to leaching of ions under the applied field gradient. Therefore, it seems that for half of the mixes, the voltage used does not cause significant damage to the specimens, and therefore influence the results. For mixes with Ms=0.45 (mixes 3, 7, 11 and 15), the voltage used in the test seems to damage the specimens, and this can be related to deficiency of silicate in the system to make a strong and dense structure. Damage to the samples from high voltage tests has been of concern with other accelerated tests [1]. More importantly, for samples with enough silica in the activator, the test seems to have not altered the ionic nature of the sample to a significant degree, and therefore the chloride penetration will be marginal as shown in Figure 9.

The chloride penetration depths of samples were measured by Vernier callipers based on the colour change boundary resulting from splitting the NT 492 test samples and spraying with $AgNO_3$, shown in Figure 8 for mixes 4 and 13 with minimum and maximum penetration depth.

Figure 9 presents the non-steady state chloride migration coefficients calculated based on the penetration depth in AA-S/F concrete mixes. The test voltage was considered to meet the maximum current limits in the standard between 10 V and 35 V and the test duration was 24 hours for all the mixes, which is the duration suggested in the standard for normal concretes. The pore solution conductivity of AA-S/F concrete is higher than that of normal concrete and the final current passed through samples was between 40 and 140 mA for different mixes, which was more than that passed through PC concrete (~50 mA for 30 V applied potential difference [20]). This can be because of pore solution conductivity in this type of concrete. The chloride penetration depth of AA-S/F concretes was between 6.09 mm to 29.10 mm for different mixes and as shown in Figure 9, the non-steady state migration coefficients for these concretes varied from 2.37 to $49.56\times10^{-12}$ $m^2/s$. Although the depth of penetration is lower, the $D_{nssm}$ is not much lower than the $D_{nssm}$ reported for PC concrete [20-21] (the chloride penetration depth of a PC concrete sample was 41.5 mm and the non-steady state migration coefficient was $3.36\times10^{-12}$ $m^2/s$ for the same test voltage and test duration).

The above non-steady state migration coefficients were calculated using the chloride ion concentration, $C_d$, provided in the standard. This value, 0.07N, is considered to be the chloride concentration at which the silver nitrate produces a white precipitate. This value is defined for PC concretes, and would not necessarily apply to the binders used in this study, however other research has shown that this figure is broadly acceptable, and possibly even more accurate for binders such as GGBS [22-23]. This would indicate that the test is suitable for AA-S/F.

The non-steady state migration coefficient, $D_{nssm}$, of the AA-S/F concretes was found to be influenced by the fly ash content, NaOH% and Ms. For NaOH% of 8% and Ms equal to 1.0, an increase in fly ash content from 20% to 80% resulted in an increase in $D_{nssm}$ while the lowest $D_{nssm}$ resulted when the fly ash content was 20%. Values of Ms of 1.0 and NaOH% of 8%, proved to be the optimum to give the lowest $D_{nssm}$.

## Correlation between chloride migration coefficient and electrical resistivity

The results show that chloride ingress in AA-S/F concretes can be predicted by bulk electrical resistivity, although the resistivity of these concretes relies on pore structure and pore solution conductivity. The non-steady state migration coefficient, $D_{nssm}$, of the AA-S/F concretes is strongly correlated to their bulk resistivity as shown in Figure 10.

# CONCLUSION

- It is possible to achieve desirable fresh properties and compressive strength for the blends studied, and all but one mix out of the 16 considered had compressive strengths over 40 MPa at 28 days. This means such concretes do not require elevated temperature for strength gain. A higher proportion of slag resulted in high early and long term strength.
- All of the AA-S/F concretes tested have moderate to high chloride migration coefficients, making them non-viable for protection of reinforcing in chloride environments.
- Variability of $D_{nssm}$ is very high between the three replicates studied. The blends can be categorised as moderate diffusivity (S/F = 80/20), high diffusivity (S/F = 60/20 & 40/60), and very high diffusivity (S/F = 20/80).

- A modulus Ms of 1.0, NaOH% of 8% and slag to fly ash ratio of 4, proved to be the optimum for low $D_{nssm}$ ($2.37\times10^{-12}$ $m^2/s$). AA-S/F concretes with more than 20% fly ash have higher $D_{nssm}$ of > $3.82\times10^{-12}$ $m^2/s$.

- The migration coefficient $D_{nssm}$, of the AA-S/F concretes is strongly related to their bulk resistivity. However, this may be unique to the set of mixes considered in this work and may not be generalised.

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**Table 1-Oxide composition of GGBS & PFA**

| precursor | Component (mass% as oxide) | | | | | | |
|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | CaO | $Fe_2O_3$ | MgO | others | LOI |
| GGBS | 35.7 | 11.2 | 43.9 | 0.3 | 6.5 | 2.09 | 0.31 |
| PFA | 46.8 | 22.5 | 2.2 | 9.1 | 1.3 | 14.5 | 3.6 |

**Table 2-Physical properties of GGBS & PFA**

| Material | GGBS | PFA |
|---|---|---|
| Fineness ≥45 µm | 7.74% | 18.39% |
| Particle density | 2.86 | 2.21 |
| Water absorption | 35.14% | 27% |

**Table 3-Physical properties of aggregates**

| Aggregates | Bulk specific gravity | Bulk SSD Specific gravity | Water Absorption (%) |
|---|---|---|---|
| Sand (0-4mm) | 2.72 | 2.73 | 0.75 |
| Fine Crushed Agg. (5-10mm) | 2.67 | 2.75 | 3.14 |
| Coarse Crushed Agg. (10-16mm) | 2.60 | 2.67 | 2.60 |

**Table 4-The details of the different mixtures and their properties**

| Mix No. | GGBS/PFA (kg/m$^3$) | GGBS/PFA ratio | Ms (=SiO$_2$/Na$_2$O) | NaOH (%) | W/B | Slump (mm) | 2 days Comp. St. (MPa) | 28 days Comp. St. (MPa) | Concrete Grades |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 340/85 | 4 | 1.0 | 6 | 0.47 | 170 | 54.6 | 81.1 | M80 |
| 2 | 340/85 | 4 | 1.25 | 6 | 0.47 | 200 | 51.2 | 84.8 | M80 |
| 3 | 340/85 | 4 | 0.45 | 8 | 0.47 | 200 | 35.2 | 60.2 | M60 |
| 4 | 340/85 | 4 | 1.0 | 8 | 0.47 | 230 | 50.9 | 80.6 | M80 |
| 5 | 255/170 | 1.32 | 1.0 | 6 | 0.46 | 230 | 42.1 | 73.4 | M70 |
| 6 | 255/170 | 1.32 | 1.25 | 6 | 0.46 | 240 | 40.3 | 78.5 | M75 |
| 7 | 255/170 | 1.32 | 0.45 | 8 | 0.46 | 205 | 28.8 | 52.4 | M50 |
| 8 | 255/170 | 1.32 | 1.0 | 8 | 0.46 | 240 | 31. 9 | 75.7 | M75 |
| 9 | 170/255 | 0.75 | 1.0 | 6 | 0.44 | 170 | 28.2 | 64.8 | M60 |
| 10 | 170/255 | 0.75 | 1.25 | 6 | 0.44 | 210 | 22.3 | 69.4 | M65 |
| 11 | 170/255 | 0.75 | 0.45 | 8 | 0.44 | 210 | 22.2 | 54.0 | M50 |
| 12 | 170/255 | 0.75 | 1.0 | 8 | 0.44 | 250 | 23.0 | 61.2 | M60 |
| 13 | 85/340 | 0.25 | 1.0 | 6 | 0.43 | 225 | 13.8 | 42.6 | M40 |
| 14 | 85/340 | 0.25 | 1.25 | 6 | 0.43 | 220 | 9.5 | 47.1 | M45 |
| 15 | 85/340 | 0.25 | 0.45 | 8 | 0.43 | 200 | 9.7 | 28.4 | M25 |
| 16 | 85/340 | 0.25 | 1.0 | 8 | 0.43 | 250 | 10.4 | 49.2 | M45 |

*Fig. 1-Cells for performing non-steady state migration test as per NT Build 492 [10].*

*Fig. 2-Bulk resistivity test equipment and sample*

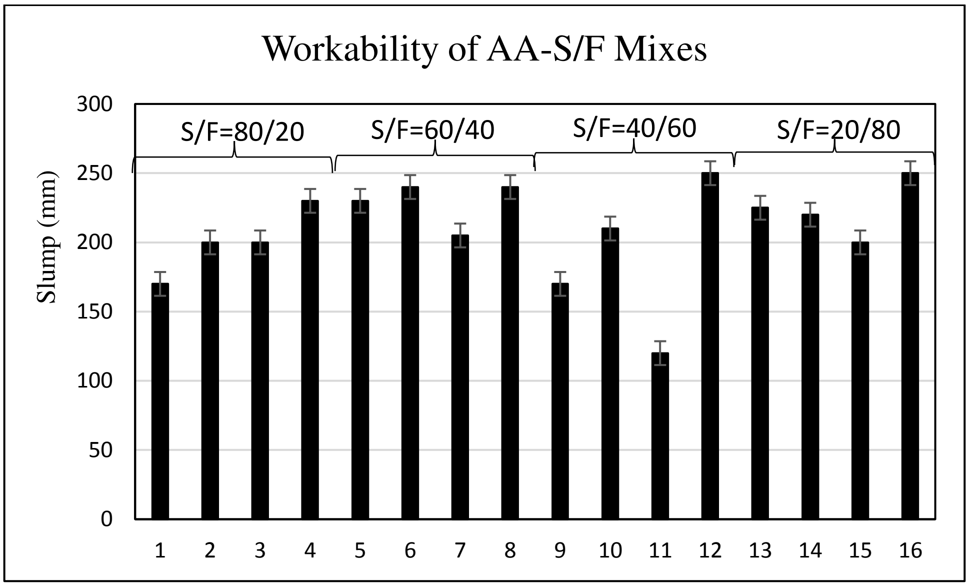

*Fig. 3-Slump results of AA-S/F concretes (S/F=GGBS/PFA ratio)*

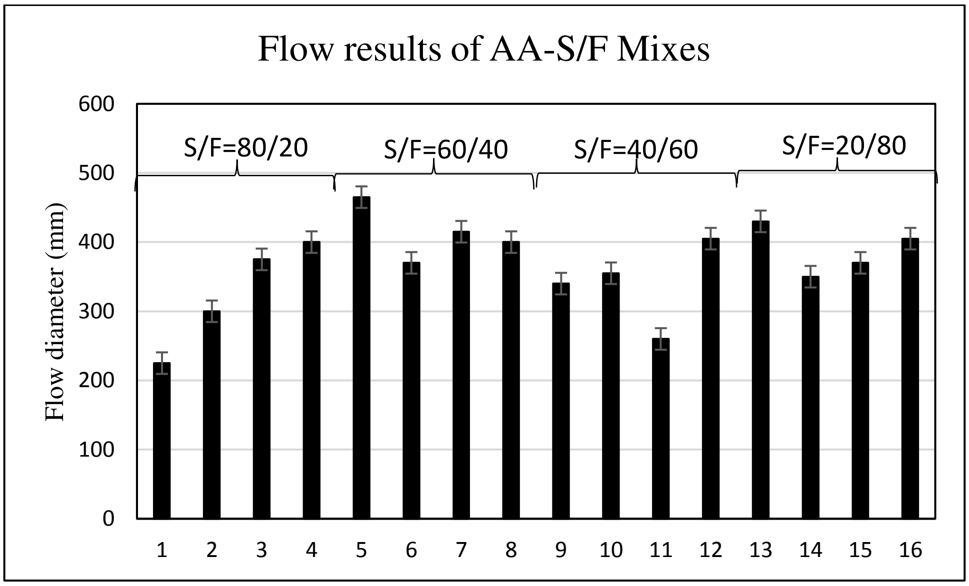

*Fig. 4-Flow results of AA-S/F concretes (S/F=GGBS/PFA ratio)*

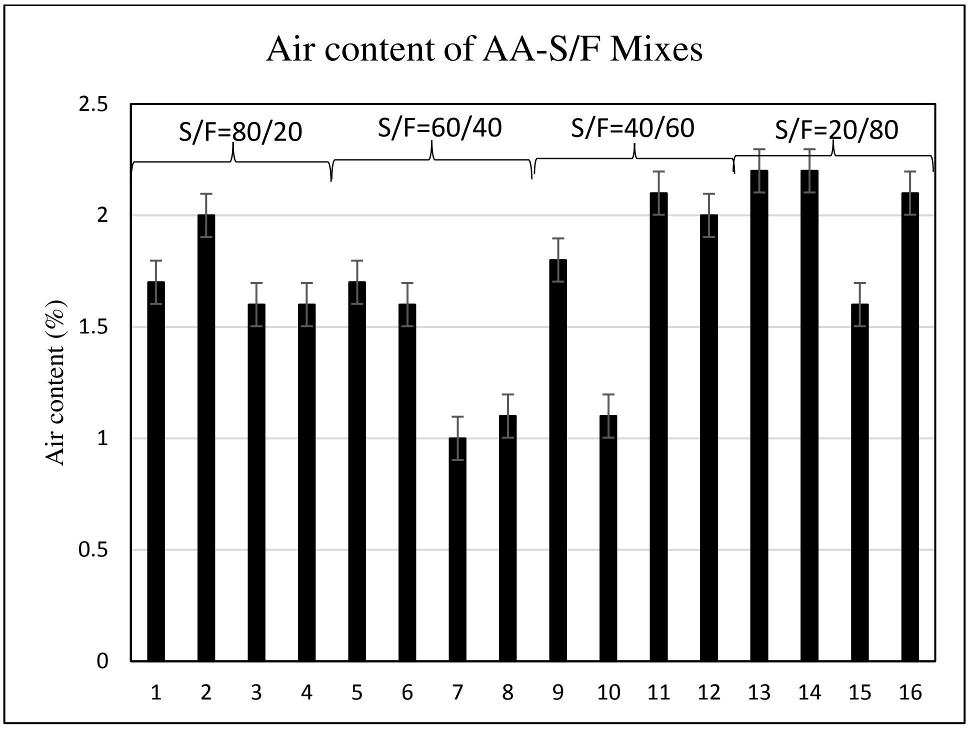

*Fig. 5-Air content in fresh mixes of AA-S/F concretes (S/F=GGBS/PFA ratio)*

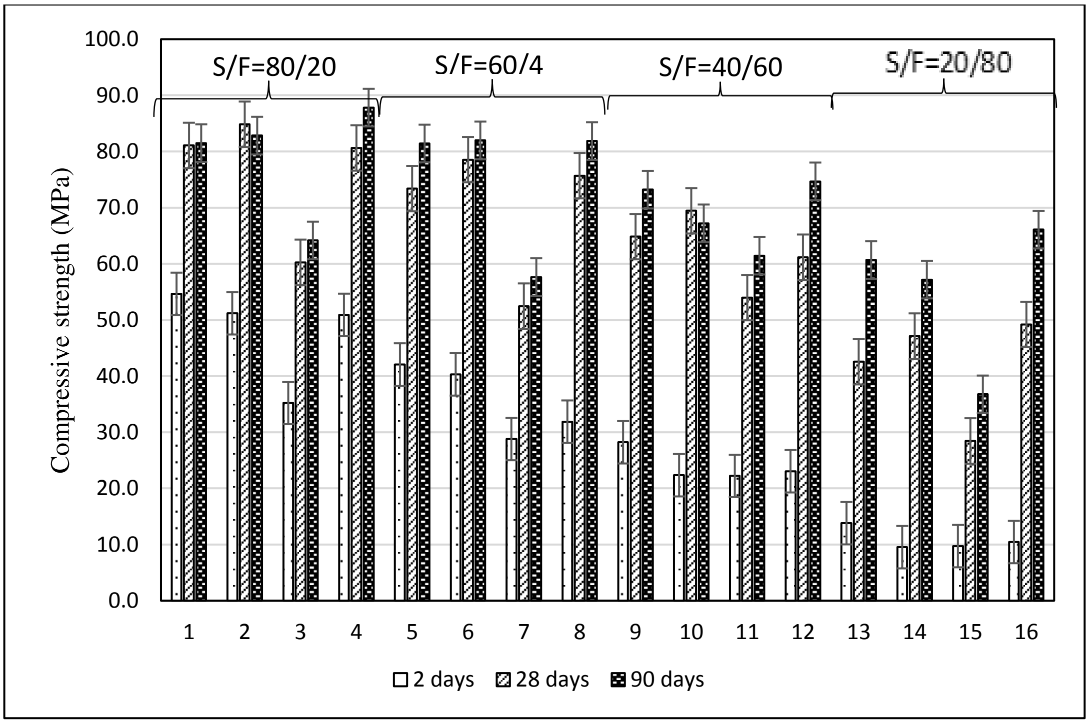

*Fig. 6-Compressive strength of AA-S/F concretes mixes at 2, 28 and 90 days (S/F=GGBS/PFA ratio)*

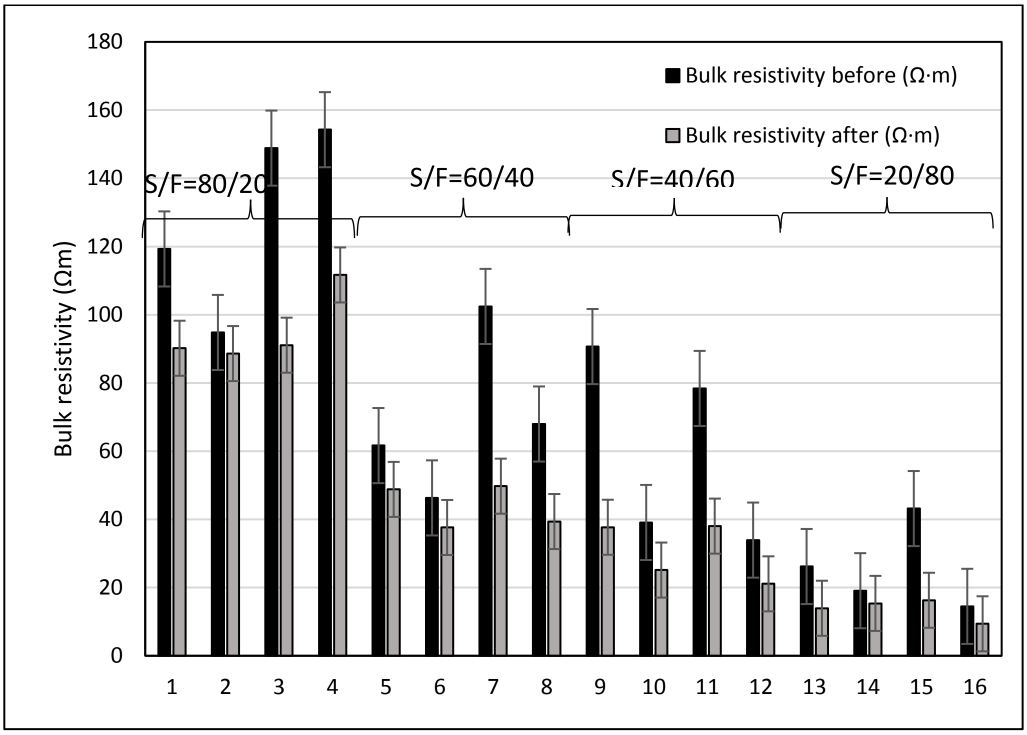

*Fig. 7-Bulk resistivity before and after migration test (NT BUILD 492)*

*(S/F=GGBS/PFA ratio)*

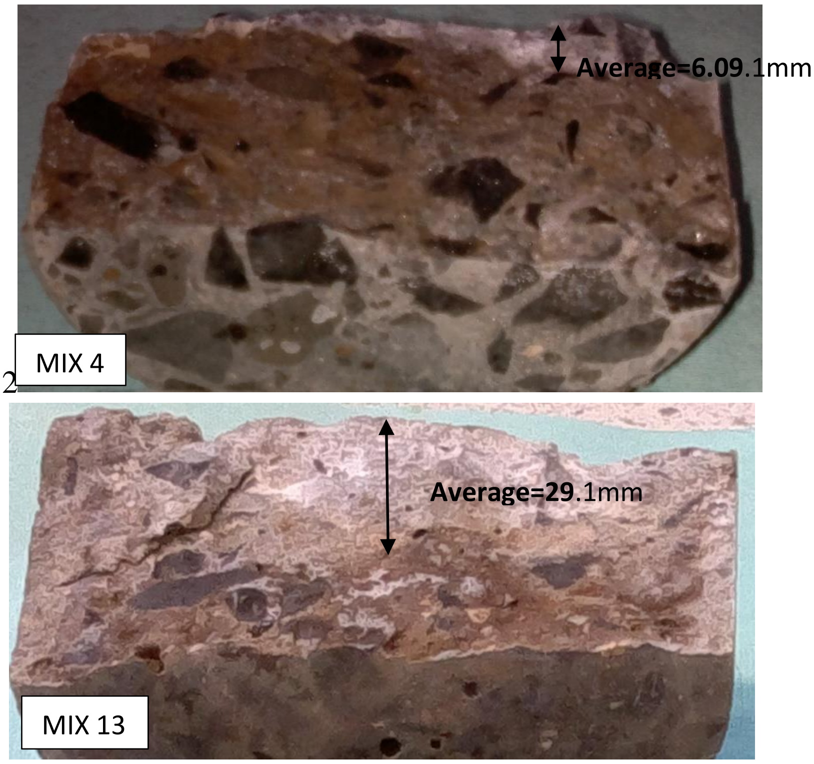

*Fig. 8-Split sample slices of concrete specimen sprayed with silver nitrate after chloride migration test (The minimum chloride penetration depth resulted for mix 4, equal to 6.09 mm and the maximum for mix13, equal to 29.10 mm)*

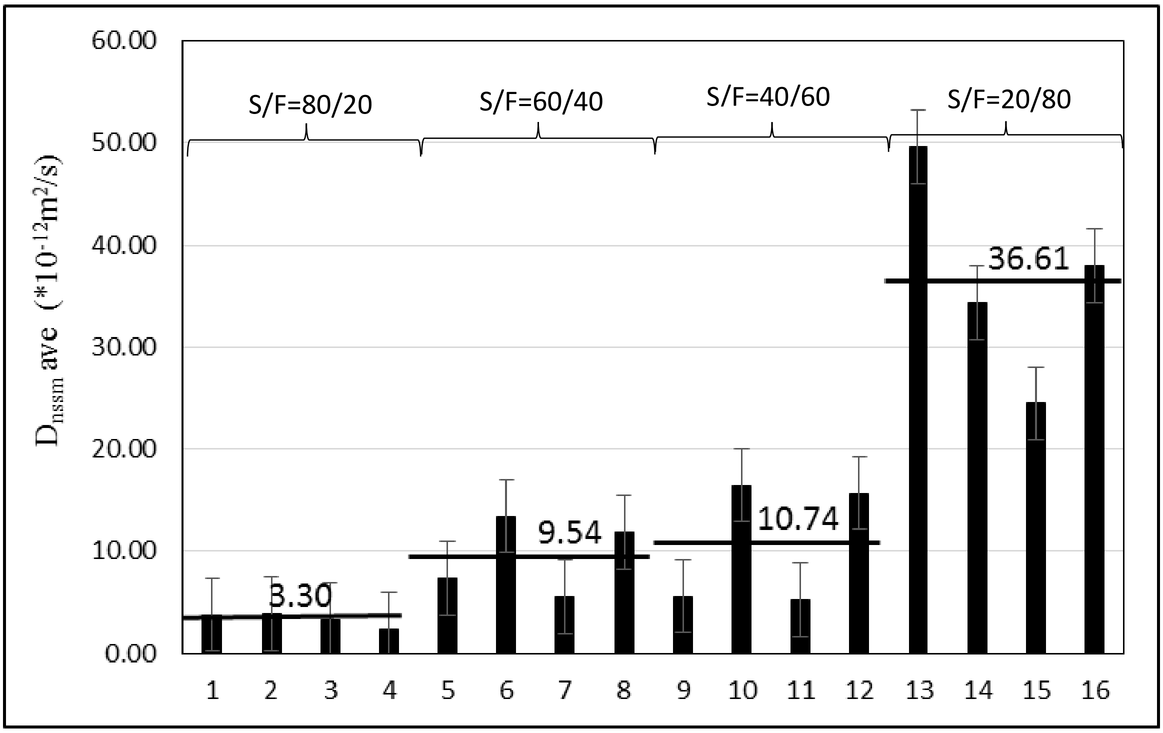

*Fig. 9- $D_{nssm}$ of the AA-S/F concrete mixes after 90 days curing determined from non-steady state chloride migration test (S/F=GGBS/PFA ratio)*

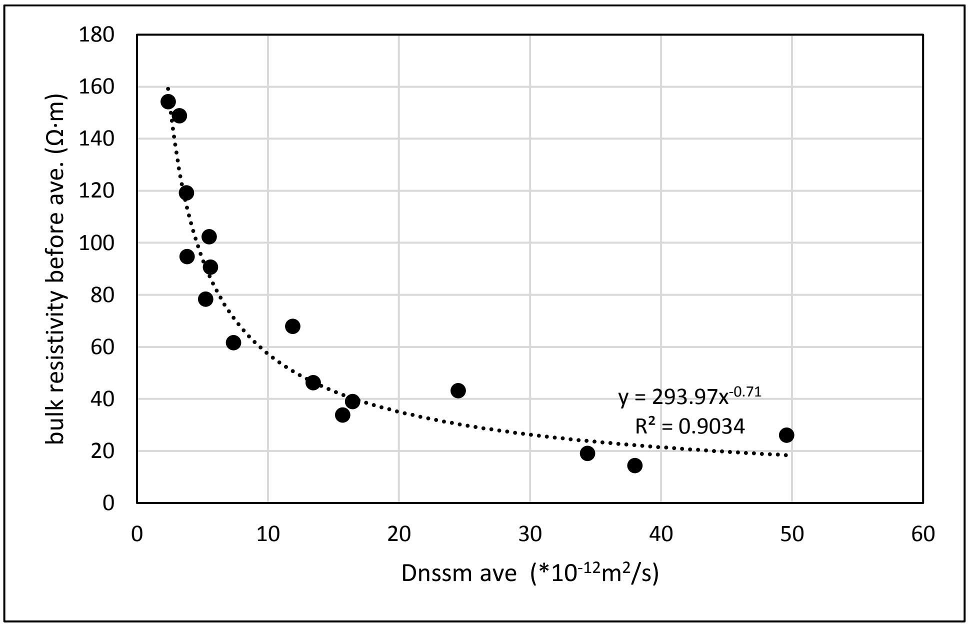

*Fig. 10-Correlation of $D_{nssm}$ and bulk resistivity of AA-S/FC*