



Correlation Between Chloride Ions' Migration and Diffusion Coefficients of Alkali-Activated Concrete

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Abstract. Alkali-Activated Materials (AAM) are considered an alternative to cementitious binders. Due to the reduction of Portland cement content and using industrial wastes to produce it, AAMs gained the researcher's interest in the last decades. Current researches show that mechanical properties and ability to ensure proper conditions for reinforcing steel of AAMs are at least as good as for Ordinary Portland Cement (OPC) concrete. However, environmental aggression affects the durability of AAMs in a different way than OPC. For this reason, there is a necessity to develop the available testing methods dedicated to cement concrete and adjust them to a new type of binder. One of the aggressive environments is a marine zone, where the chloride ions affect the properties of structures. In this paper, the comparison of results obtained from chloride penetration tests conducted on three Alkali-Activated Concrete (AAC) mixtures will be presented. The binders' precursors are the blends of Fly Ash (FA) and Ground Granulated Blast Furnace Slag (GGBFS) in slag proportions: 5%, 20%, and 35% expressed by the mass of FA. Materials are denominated AAC5, AAC20, and AAC35, respectively. Chloride penetration tests were conducted using the modified NT BUILD 492 rapid migration test, and reference ASTM C1556 chloride diffusion test. The correlation of results shows the necessity of applying the adjustments to the rapid migration test for AACs. In both tests, the positive influence of increasing slag content was determined. The possible reasons for the observed tendencies are presented and explained.

Keywords: Alkali-Activated Concrete · chloride penetration · NT Build 492 · ASTM C1556 · RCPT

1 Introduction

Alkali-Activated Concrete (AAC) became an alternative to ordinary Portland cement concrete [1]. This is due to the move toward the reduction of CO₂ emissions. Production of Portland cement contributes between 5% to 7% of anthropogenic CO₂ emissions

worldwide [2]. Cement production needs to be reduced to meet the requirements of current changes in emission standards [3]. Production of AAC requires an alkaline solution as an activator and aluminosilicate precursors such as fly ash (FA), ground granulated blast-furnace slag (GGBFS), metakaolin, or others. To obtain the precursors, industrial by-products are utilized what reduce CO₂ emissions associated with unnecessary Portland cement production. The process called geopolymerization in origin was described by Davidovits [4]. Decades of investigations increased the knowledge level, however, the application of Alkali-Activated Materials in construction is still very limited [5]. These materials exhibit magnificent binding properties, thermal and chemical resistance [6, 7] in comparison to ordinary Portland cement concrete. The final properties of Alkali-Activated Materials are related to the ingredients used, the procedure of mixing, curing conditions, the SiO₂/Al₂O₃ ratio, and other factors [8]. The reaction of geopolymerization depends on the reactivity of the precursor in an alkaline environment that is related to the content of amorphous phases and their compositions. Higher content of vitreous phases means a higher degree of reactivity and quicker activation [9]. GGBFS is well known for a high amount of amorphous phases rich in CaO, SiO₂ and Al₂O₃ that enables a process of hardening in ambient temperature [10, 11]. In the case of FA, the compositions are very variable which causes difficulties in the comparison of results from different studies [7]. The most popular for geopolymer concrete preparation are class F fly ashes. They consist of amorphous and crystalline phases like quartz, mullite, iron oxides, and lime [12–14]. Due to the low calcium content activation process requires additional temperature curing between 40 °C and 85 °C [15, 16]. The next step on the way to development was research on blended precursors. As a result, the innovative materials harden in the ambient temperature conditions [17–20] as also show great mechanical properties and resistance to aggressive environments [21, 22]. The properties of blended binders are very variable in the case of precursors used and their mixtures. In this paper, the properties and resistance to chloride aggression of blends of FA and GGBFS as the precursors for AACs are investigated.

Chloride penetration into concrete can be caused by capillary absorption, hydrostatic pressure, and diffusion [23]. The first of them is driven by moisture gradients. When a solution containing chloride ions meets a dry surface of concrete it will be taken into the pore structure by capillary suction. This mean of chloride penetration is common but the penetration depth caused by absorption is minor when compared to other mechanisms. Hydrostatic pressure takes place when the gradient of pressure appears. The hydraulic head containing chloride ions applied on a concrete surface can result in the permeation of solution into the pore structure. In fact, the most influencing mechanism of chloride penetration is diffusion. It appears with chloride ions concentration gradient. Diffusion of chloride ions into the concrete is considered a main mechanism of chloride aggression for this type of material and is widely investigated to describe resistance to chloride penetration.

There are variety of methods to measure resistance to chloride aggression [23, 24]. The AASHTO T259 test called the “salt ponding test” involves the exposure of the top face of the sample to a 3% NaCl solution for 90 days. The bottom face of a sample is exposed to drying conditions as depicted in Fig. 1.

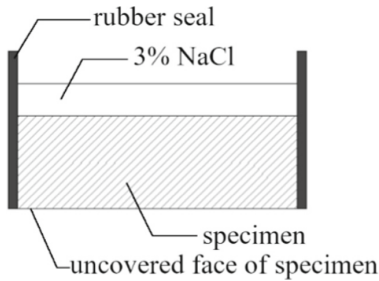


Fig. 1. Scheme of AASHTO T259 test.

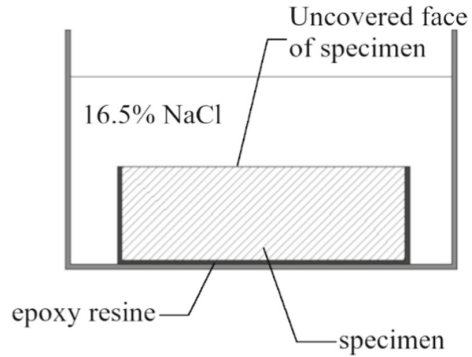


Fig. 2. Scheme of ASTM C1556 test.

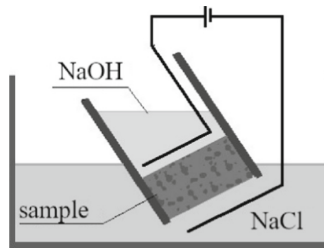


Fig. 3. Scheme of accelerated chloride migration test

The most reliable test is ASTM C1556 [25] (or equivalent NT Build 443 [26]) called the Bulk diffusion test (BDT). The method consist in exposure of the sample with uncovered top face (other sides of sample are coated with epoxy resin) to aggressive 16.5% NaCl solution. The scheme of ASTM C1556 is presented in Fig. 2.

Both the tests and the analysis of their results are time-consuming. This is why most of the researchers prefer accelerated methods. This group of tests relies on an application of an external potential across the specimen and forcing the chloride ions outside to migrate into the specimen (see Fig. 3). After the test specimen is sprayed with silver nitrate solution and the results are obtained by the visual analysis of chloride penetration depth. This group of tests contains NT Build 492 [27], EN 12390-18 [28], Perfdub French national project recommendations [29], and more available instructions [24, 30, 31]. Application of external potential to the sample results in a short time of test duration (6 h–96 h) which makes it more suitable for researchers. On the other hand, rapid tests were invented for cement concretes that have lower electrical conductivity than AAC. Some of the researchers use rapid testing procedures for AACs [30, 32, 33], but due to the difference in electrical conductivity, this approach needs more attention than reference tests and probably some modifications. In this paper, the comparison of results obtained from reference test ASTM C1556 and modified NT Build 492 are compared and analysed.

2 Materials

The objects of this investigation were six compositions of Alkali-Activated Concretes. They were composed of Fly Ash (Połaniec powerplant, Poland) with Ground Granulated Blast Furnace Slag (Ekocem Dąbrowa Górnicza, Poland) replacement by mass. Three levels of GGBFS were tested: 5%, 20%, and 35%. The blends were denominated AAC5D, AAC20D, and AAC35D for dolomite and AAC5B, AAC20B, and AAC35B for basalt aggregate. The names of materials contain the number that means the GGBFS content. The compositions of tested mixes and main properties of hardened materials are presented in Table 1.

Table 1. Compositions and main properties of Alkali-Activated Concretes [kg/m³]

[kg/m ³]	AAC5D	AAC20D	AAC35D	AAC5B	AAC20B	AAC35B
FA	336.9	292.3	244.1	336.9	292.3	244.1
GGBFS	17.7	73.1	131.4	17.7	73.1	131.4
Alkaline Solution + water	189.4	195.1	200.5	189.4	195.1	200.5
Sand 0/2	663.6	663.6	663.6	662.4	662.4	662.4
Dolomite 2/8	644.6	644.6	644.6	0.0	0.0	0.0
Dolomite 8/16	589.6	589.6	589.6	0.0	0.0	0.0
Basalt 2/8	0.0	0.0	0.0	708.9	708.9	708.9
Basalt 8/16	0.0	0.0	0.0	648.4	648.4	648.4
Density [g/cm ³]	2.20	2.29	2.32	2.29	2.36	2.40
f _{c28} [MPa]	23.5	40.4	58.4	26.0	42.1	60.3
f _{c180} [MPa]	38.3	56.6	68.0	40.8	51.0	68.2

Fly Ash used to prepare concretes was categorized as class F according to ASTM C618 [34]. Silicon dioxide and aluminum oxide are the main components of FA. GGBFS was mainly composed of calcium oxide and silicon oxide. Precursors' oxide compositions are shown in Table 2.

Table 2. Chemical composition of FA and GGBFS used.

wt.%	SiO ₂	Al ₂ O ₃	Fe _x O _y	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	P ₂ O ₅	TiO ₂	Mn ₃ O ₄	Cl ⁻
FA	52.30	28.05	6.32	3.05	1.71	0.28	2.51	0.76	0.69	1.35	0.07	-
GGBFS	39.31	7.61	1.49	43.90	4.15	0.51	0.36	0.47	-	-	-	0.04

The activator used for blends was a diluted sodium silicate solution Geosil® 34417, supplied by Woellner. Specifications of the product declared by the producer are presented in Table 3.

Table 3. Composition of Geosil® 34417

Characteristic	Unit	Woellner Geosil® 34417
Na ₂ O content	wt. %	16.74
SiO ₂ content	wt. %	27.5
Density	g/cm ³	1.552
Viscosity	mPa × s	470
Weight ratio (WR = wt. % SiO ₂ /wt. Na ₂ O)	-	1.64
Molar ratio (MR = mol SiO ₂ /mol Na ₂ O)	-	1.70

The mixing procedure was based on the experience of the research team [35]. Firstly, the pasted were prepared. A diluted alkaline solution was mixed with FA to obtain a homogenous consistency. The stirring process was not stopped to prevent the formation of lumps when GGBFS was added. Parallely, a quartz sand and coarse aggregate were mixed in the second, larger mixer. When the paste was homogenized, it was slowly poured into the large mixer with the aggregate. All ingredients were mixed until the paste was well distributed in the mass. The samples were cast in cylindrical moulds Ø11 × 22 cm and compacted on a shrinking table. The specimens were kept in ambient conditions (temp. 20 ± 2 °C) with protection from water evaporation (covered by the plastic film). Thermal curing was not applied. Most of the specimens were removed from moulds after 1 day. The specimens of concrete with 5% of GGBFS content were demoulded after 2 days.

3 Methods

3.1 ASTM C1556

Investigation of the chloride diffusion coefficient was performed ASTM C1556 test method. The test specimens were Ø11 × 5 cm cylinders cut from Ø11 × 22 cm cylinders at the age of 360 days. After the preparation of the samples (coverage by epoxy resin and saturation with distilled water), they were placed in the test cell fulfilled with 16.5% aqueous NaCl solution for 35 days. On completion of immersion, the specimens were removed from the solution and kept for 24 h in laboratory conditions. Then the specimens were ground using Germann Instruments' Profile Grinder to obtain the powder samples. The maximum depth of grinding was 50 mm in layers parallel to the exposed surface. For each layer, the weight of the powder sample was at least 10 g.

Investigations of the acid-soluble chloride contents of the powder samples were performed according to ASTM C1152 [36] method with use of 5 g instead of 10 g recommended. The volume of a solution prepared for each powder sample was 250 ml. From each of the solutions, 2 samples of 50 ml were tested and the average value of chloride content was calculated with the use of a potentiometric titration machine. As recommended in ASTM C114 [37], a silver nitrate solution was used to determine the chloride content. According to ASTM C1556, the values of apparent chloride diffusion

coefficient (D_a) and surface concentration of chloride ions (C_s) were determined by fitting the Eq. 1 to the chloride profile by the means of non-linear regression analysis using the method of least squares.

$$C(x, t) = C_s - (C_s - C_i) \times \operatorname{erf}\left(\frac{x}{\sqrt{4 \times D_a \times t}}\right) \quad (1)$$

where:

$C(x, t)$ chloride concentration, measured at depth x and exposure time t , mass %,

C_s projected chloride concentration at the interface between the exposure liquid and test specimen that is determined by the regression analysis, mass %,

C_i initial chloride-ion concentration of the cementitious mixture prior to submersion in the exposure solution, mass %,

x depth below the exposed surface (to the middle of a layer), m,

D_a apparent chloride diffusion coefficient, m^2/s ,

t the exposure time, s,

erf the error function.

To fit the Eq. (1) to experimentally obtained points MATLAB® code was used.

3.2 NT Build 492 Test

This procedure was used for rapid determination of chloride ions penetration into the concrete for non-steady-state conditions. Application of voltage enabled to shorten the test duration, however, as was explained before, the electrical conductivity of tested material strongly affects the obtained results. In this study testing procedure was modified, as it was proposed by Noushini and Castel [38] for the ASTM C1202 test. The voltage applied during the test was reduced from the original 60V to the value of 10V. Due to high electrical conductivity, a lower voltage of 10 V was applied rather than the standard 30 V (maximum 60V). The initial values of current were noted and used to select the time of the test according to the standard's recommendations. The duration of the tests was 24 h for materials with 20% and 35% of slag and 6 h for 5% GGBFS content.

The solutions used were 10% NaCl (catholyte) and 1.2% NaOH (anolyte). The specimens were vacuum saturated with distilled water before the test. After completing the tests, samples were split in half and sprayed with silver nitrate solution that becomes a grey sediment in combination with chloride ions. Chloride penetration depths (x_i) were measured with 10 mm intervals and the average value (x_d) was included in the calculation of the non-steady-state migration coefficient (see Eq. 2).

$$D_{nssm} = \frac{R \cdot T}{Z \cdot F} \cdot \frac{e}{\Delta E} \cdot \frac{x_d - \sqrt[4]{x_d}}{\Delta t} \quad (2)$$

where:

D_{nssm} non-steady state migration coefficient, m^2/s ;

Z absolute value of ion valence, for chloride, $z = 1$;

F Faraday constant, $F = 9.648 \times 10^4 \text{ J}/(\text{V} \cdot \text{mol})$;

ΔE value of applied voltage, V;

R gas constant, $R = 8.314 \text{ J}/(\text{K} \cdot \text{mol})$;

T average value of the initial and final temperatures in the anolyte solution, K;

e thickness of the specimen, m;

X_d average value of the penetration depths, m;

Δt test duration, seconds;

erf^{-1} inverse of error function;

C_d chloride concentration at which the colour changes, $c_d \approx 0.07$ for OPC concrete;

C_0 chloride concentration in the catholyte solution, N.

The rule of chloride penetration depths measurements of a split sample is presented in Fig. 4.

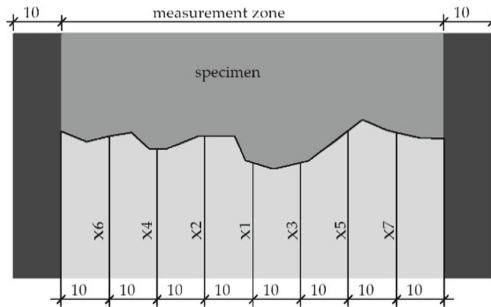


Fig. 4. The rule of chloride penetration depths measurements

4 Results and Discussion

4.1 Astm C1556

The first reason for conducting this investigation was to determine the values of chloride diffusion coefficients for Alkali-Activated Concretes with variable binders' compositions and basalt aggregate. The results of performed tests enabled to show the profiles of chloride content in the materials. Chloride profiles of AACs and the curves fitted (see Eq. (1)) to experimentally obtained points are presented in Figs. 5a, b, and c.

Results depicted in Figs. 5a, b, and c show a positive effect of GGBFS addition on chloride penetration depth. The specimen with 5% slag content exhibits the highest chloride ions concentration. Surface chloride concentration determined by curve fitting is 0.61% (percentage of chloride ions mass in the mass of powder sample) while for AAC20 and AAC35 the values are 0.49% and 0.54% respectively. For the concretes with the highest GGBFS content the concentration of chlorides decreases rapidly and reaches 0.13% in the fifth layer (17.5 mm depth). In case of low slag materials, the decrease of chloride ions is slower. For AAC20 and AAC5 values of Cl⁻ concentrations reached values of 0.13% only at the depths of 32 mm and 43.5 mm respectively.

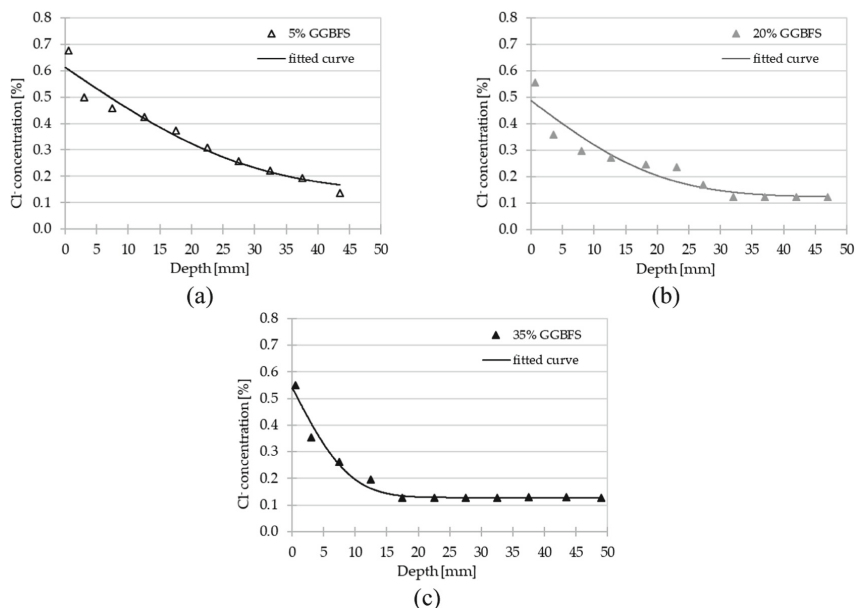


Fig. 5. Chloride profile of AACs and curves fitted to the experimental results: (a) AAC5, (b) AAC20, (c) AAC35.

4.2 NT BUILD 492

After completing NT BUILD 492 tests the samples were split and sprayed with silver nitrate solution. The examples of graphic analysis of chloride penetration depths for AAC5D, AAC20D, and AAC35D are presented in Fig. 6.

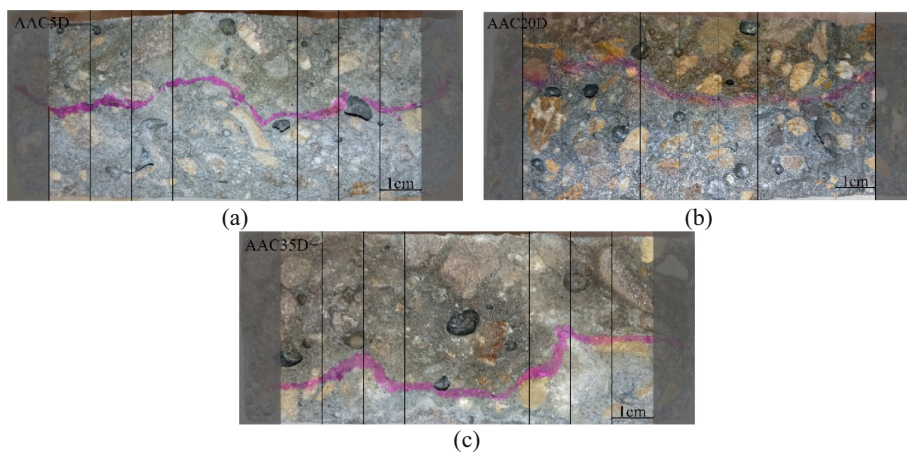


Fig. 6. Chloride penetration depths: (a) AAC5D – 6 h, (b) AAC20D – 24 h, (c) AAC35D – 24 h.

One of the difficulties in performing the NT BUILD 492 test is reading the results correctly. Silver nitrate solution reacts with chloride ions and a grey precipitate is formed. However, the colour of the mortars based on FA and GGBFS is strongly related to the proportions of ingredients. In case of low GGBFS content (AAC5), the colour of non-infected mortar is very similar to the colour of the precipitation appearing after silver nitrate spraying. For this reason, the evaluation of chloride penetration depths in Alkali-Activated Materials is particularly problematic.

In Table 4 the values of chloride ions migration coefficients for different GGBFS contents obtained using NT BUILD 492 method for concretes with basalt and dolomite aggregates are presented.

Table 4. The values of chloride ions migration coefficients obtained using NT BUILD 492 method [$\times 10^{-12}$ m/s²]. B – basalt aggregate; D – dolomite aggregate

AAC5B	AAC5D	AAC20B	AAC20D	AAC35B	AAC35D
82.85	81.55	40.49	40.07	13.41	10.19
96.20	97.06	44.23	40.36	15.09	15.13

Comparing the results for the same GGBFS content shows that the influence of used aggregate on the values of migration coefficient for concretes was negligible. This results show clearly that the chloride ions penetration takes place mainly in the binder.

4.3 Chloride Ions Diffusion and Migration Coefficients Correlation

The values of chloride migration coefficients obtained from the NT BUILD 492 test compared to values of apparent chloride diffusion coefficient are presented in Fig. 7. The average values of migration coefficients are depicted as triangle points.

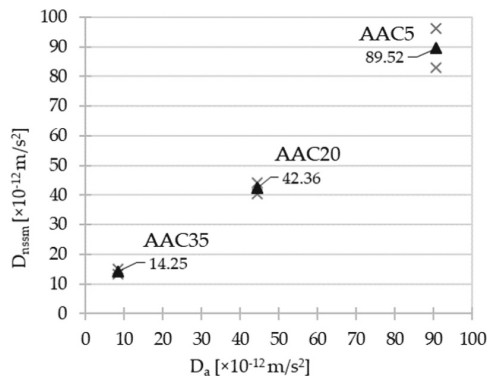


Fig. 7. The correlation between values of chloride ions diffusion and migration coefficients obtained using ASTM C1556 and NT BUILD 492 methods [$\times 10^{-12}$ m/s²].

According to NT BUILD 492, the coefficient of variation of reproducibility for concrete mixed with slag cement is 24%. The ratios of the difference values to the average values of coefficients for each of the tested materials did not exceed the value of 24%. The values of D_a decrease with increasing GGBFS content the same as it was presented for BDT results.

The important point of this investigation was to verify if there is a necessity for a change of voltage applied to Alkali-Activated Concretes during the accelerated chloride migration tests. Noushini and Castel [38] presented the modification of voltage applied in the ASTM C1202 [40] test. The standard voltage of 60 V was reduced to 10V which was found to be more suitable for AAC. The results showed a great correlation with the reference test ASTM C1556. The same reference method was investigated and compared to NT BUILD 492 test. Both rapid tests (ASTM C1202 and NT BUILD 492) are based on the application of voltage to the specimens to accelerate the chloride ions migration into concrete, however, their procedures and output data are various. Values obtained experimentally from BDT and NT BUILD 492 for AAC with basalt aggregate are presented in Table 5.

Table 5. The mean values of chloride ions diffusion and migration coefficients obtained using ASTM C1556 and NT BUILD 492 methods [$\times 10^{-12}$ m/s²].

	AAC5B	AAC20B	AAC35B
ASTM C1556	90.67	44.38	8.55
NT BUILD 492	89.52	42.36	14.25

The values of chloride diffusion and migration coefficients for the same material obtained by the two methods are sufficiently close. For AAC35B the value of D_a calculated with the reference method is 8.55×10^{-12} m/s² while the average value of 2 specimens tested according to modified NT BUILD 492 is 14.25×10^{-12} m/s². For AAC20B and AAC5B the values of effective diffusion coefficients are 44.38×10^{-12} m/s² and 90.67×10^{-12} m/s² respectively. For these concretes average values from the rapid test are 42.36×10^{-12} m/s² and 89.52×10^{-12} m/s². Castellote et al. [41] explained that results obtained from different methods should not be directly compared. Values obtained by ASTM C1556 (steady-state conditions) are calculated without correction for chloride binding. The result of NT BUILD 492 is a non-steady-state migration coefficient value that takes into account chloride binding. The results presented in the Table 5 show a minor difference in values of the coefficients for AAC5B and AAC20B, however, the difference between migration and diffusion coefficients for material with higher strength (AAC35B) was more significant and reached 5.70×10^{-12} m/s².

5 Conclusions

Results of this investigation enabled to formulate the following conclusions:

- good correlation between the non-steady-state migration coefficient and chloride diffusion coefficient for Alkali-Activated Concretes with FA/GGBFS binders.

- nature of aggregate (basalt and dolomite) was found to have a negligible impact on the values of chloride migration coefficients.
- values of chloride migration coefficient tested in non-steady-state conditions show high convergence with the chloride diffusion coefficient tested in steady-state conditions.
- values of coefficients for materials with 5% and 20% of GGBFS content are almost identical for both ASTM C1556 and NT BUILD 492 methods. This may suggest a minor impact of chloride binding on the values of coefficients for Alkali-Activated Concretes.
- Alkali-Activated Concretes with high GGBFS content present low diffusion and migration coefficient values. Nevertheless, the dispersion of obtained results was higher than for AAC with lower GGBFS ranges and lower strengths. This aspect needs to be investigated in depth in further research.

As the results have shown, the nature of aggregates (basalt, dolomite) does not influence migration coefficient values. Nevertheless, similar research for other types of aggregates - riverbed and calcareous should be continued, and the role of ITZ (interfacial transition zone) in chloride ions movement needs to be established.

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