Geopolymers

DURABILITY PERFORMANCE OF ALKALI-ACTIVATED METAKAOLIN, SLAG, FLY ASH, AND HYBRIDS

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ABSTRACT

The durability performance of Alkali-Activated Cements (AAC) i.e., metakaolin (AAMK), slag (AAH), fly ash (AAB), and their hybrids (AAMK/H, AAMK/B) were carried out by a carbonation test and degradation test in sulfuric acid pH 0.5. The effect of carbonation was followed by IR absorption spectroscopy, KBr method. Ordinary Portland Cement (OPC), modified-OPC, and AAC were monitored for 11 weeks. Series of AAMK, AAH, AAB, and their hybrids were prepared using 2 types of alkaline activator (K0.5 and K0.64) and their deterioration were monitored for 9 weeks. Any accelerated carbonation could be ruled out in the case of AAMK, while it occurred the more the higher the CaO content of the raw material. In those materials carbonation occurred with the possible formation of vaterite which split in the IR bands at about 1420 and 1490 cm^{-1} before exposure. Further reaction was detected by increasing the intensity of vaterite phase after being exposed to CO_2 of 3 vol%. For the sulfuric acid, the AAC revealed much better compared to OPC. A comparison between 2 types of alkaline activator revealed that higher alkaline content yielded more deterioration. As compared between sources of starting materials, AAB tended to resist acid attack better than did AAMK. However, the AAB showed significant formation of cracks. The results are also discussed with respect to their long term mechanical properties followed over 5 years.

INTRODUCTION

The most important aspects of materials used in the cement and concrete industry are not only the mechanical strengths, but also that of the probability of enhanced durability. The equivalent performance to OPC is one target for AAC, but, in many cases, the properties of AAC actually are superior [1]. This binder is promoted to the industry with its outstanding properties in various fields of applications [2].

This material was originally developed from local soils and compounds of the alkali metals, i.e., sodium or potassium, so-called Soil Silicates [3, 4]. The soil-silicate concretes are highly impervious have high ratings of wear ability approaching that of granite, good cohesion with the steel reinforcement, and also resistance in waters with low bicarbonate hardness and in mineralized magnesium sulfate waters as well as sea water. This concrete has been applied for hydraulic and irrigation structures by the Ministry of Land Reclamation and Water Economy of Ukraine SSR since 1962 [4].

Based on the same theory of this binding system with further variation of starting materials used, the slag-alkaline cement was promoted by its high strength and economical aspects for an application as building materials [5, 6]. Regarding the durability performance, the slag-alkaline concrete were used with the best advantage in structures under cold temperature conditions at -15℃ to -30℃, and resisted exceeding 1,000 cycles with no expansion as a result of sudden freezing of the undercooled water. Frost destruction of the slag-alkaline stone occurred, mainly, upon cooling down to -40℃ and below.

The other materials in this family based on aluminosilicate binders termed polysialate or so-called geopolymer, were introduced and first patented in 1972 [7]. This binder was manufactured for various types of industrial products, e.g., a new class of special cements and blended cements: PYRAMENT® cements, TROLIT® and WILLIT® binders, GEOPOLYMITE® resin binders, and ceramic composites GEOPOLYCERAM®. These industrial products could be applied in a wide range of applications due to their excellent properties, e.g., ceramic-type materials under low temperature manufacturing, toxic waste management, hard surfaces, and thermal stability [2, 8].

Recently, MC-Bauchemie Müller GmbH & Co. KG, Germany [9] launched a new product named Konusit™ claimed to be a mineral based, polymer silicate bond coat. It is available in both powder and liquid forms consisting of: Konusit HB, Konusit KK 10, Konusit KK 20, Konusit KK 30 and Konusit NB as a curing agent. These products are advertised as a cement-free, environmentally friendly, very high chemical resistant against numerous acids and solvents in the range of pH = 0 to pH = 8 (but not hydrofluoric acid), impermeable to liquids, very good adhesion on mineral-based substrates (concrete, brickwork), high resistance against mechanical impact, abrasion and high temperatures, and easy application with brush or paint-brush, hand- and spray applications. In Australia, Zeobond Pty Ltd. [10] was founded by Prof. Jannie S. J. van Deventer launched a new product named E-Crete™ for construction applications. This material provides significant environmental savings with technical superiority in terms of fire and chemical resistance.

It is important for assuring long-term durability to characterize more fully the complex solid phases, including determining the combined state of alkali in the solid hydration products, and of the residual soluble species in the pore fluids as a function of time [1]. As a consequence of those excellent properties and potential candidates for applications under extreme conditions of this material, a series of AAC in paste and mortar was subjected to accelerated carbonation testing and degradation testing in sulfuric acid (pH 0.5). This pilot study was taken to assess durability and performance of the specimens in this series of starting materials.

EXPERIMENTAL PROCEDURES

Fly ash (Brown coal fly ash ‘B’: Mae-Moh Power Plant, Thailand), ground granulated blast furnace slag (Hüttensand ‘H’: Holcim GmbH, Germany) and metakaolin (‘MK’: MC-Bauchemie Müller GmbH & Co., Germany) were used as aluminosilicate source materials. The chemical composition of the solid materials is given in Table 1. In order to make the K-based alkaline activator, KOH pellets (Carl Roth GmbH, Germany) were dissolved in distilled water to 8M concentration and mixed with potassium silicate solution ($SiO_2:K_2O \approx 2.5$, molar ratio: Woellner GmbH & Co. KG, Germany) in 3 series using $K_2O/SiO_2 = 0.5$, 0.6 and 0.64 molar ratio, i.e. K0.5, K0.6 and K0.64, respectively. The mixture of alkaline activator was prepared 1 day before mixing. The fly ash (B) and slag (H) were partially replaced with 0, 10, 30 and 50 wt% metakaolin (MK), mixed and then alkali-activated in portions as given in Table 2.

Table 1. Chemical composition of solid materials (wt%).

Oxide component	SiO_2	Al_2O_3	CaO	Fe_2O_3	TiO_2	Mn_2O_3	MgO	K_2O	Na_2O	SO_3	LOI
B	45.89	24.58	10	10.67	0.46	0.08	2.44	2.65	1.32	1.76	1.11
H	36.52	10.17	41.61	0.32	0.51	0.15	5.43	1.06	0.64	0.38	0.81
MK	49.92	41.45	0.03	0.32	0.47	0	0.01	0.14	0.26	0.06	0.52

The AAC specimens were divided in 2 types: cement paste (for chemical analysis) and cement mortar (mixture of cement paste with standard sand) for mechanical investigation and corrosion observation. The alkaline solution to aluminosilicate source materials ratio was maintained at 0.5 for K0.5, K0.6 and K0.64 series of fly ash, slag and hybrids. For the pure metakaolin (AAMK) series, the mixture contained a higher content of alkaline solution of 1 as shown in Table 2. The K-based alkaline activator was diluted with distilled water for improving the workability of the matrix.

Table 2. Mixture proportion of alkali-activated metakaolin (AAMK), fly ash (AAB), slag (AAH) and hybrids in weight ratio.

Mixture	Solid materials		K-based activator	Distilled water
	B or H	MK		
AAMK	-	1	1	0.25
AAB or AAH	1		0.5	0.25
Hybrids : AAMK/B Fly ash based or AAMK/H Slag-based	0.9	0.1	0.5	0.25
	0.7	0.3	0.5	0.25
	0.5	0.5	0.5	0.25

For chemical analysis, i.e. FTIR absorption spectroscopy (KBr method), the cement mixtures were prepared separately in the same proportion as described in Table 2. The specimens were cast in a closed plastic box and then cured at room temperature (20±2°C) with a relative humidity of not less than 50% until they were tested at specified times.

Table 3. Mixture proportions of OPC, modified-OPC, and AAC mortar prisms.

	Symbol	CEM I 42.5R (g)	B (g)	H (g)	MK (g)	K0.5 (g)	Water (g)	Standard sand (g)
OPC	C1	450	-				225	1350
Modified OPC	M1	270	90	90		-	157.5†	1350
	M2	270	90	-	90	-	157.5†	1350
	M3	180	90	90	90	-	157.5†	1350
AAC	G1	-	270	90	90	225	45	1350
	G2	-	180	90	180	247.5	67.5	1350
	G3	-	135	90	225	270	67.5	1350
	G4		90	90	270	292.5	67.5	1350
	G5§		45	90	315	292.5	67.5	900‡

Remark: All data in this Table corresponds to the specimens in Figure 2.
Age of specimens: C1 = 9 months, the others = 2 weeks immersed in water at 20°C before corrosion testing.
§ G5: a coating layer, was applied for all specimens in Figures 2(b) and 2(c).
† Superplasticizer: Worement FM265 was added 2 wt% of binder.
‡ River sand: Washed river sand (0.5–2 mm).

The mixture proportion of OPC, modified-OPC, and AAC mortar prisms is presented in Table 3. The procedures of mixing and moulding mortar specimen were prepared conforming to the European standard EN 196 Part 1 into 4 cm x 4 cm x 16 cm standard prism. The proportion by mass of mortar composed of one portion of solid materials, three portions of sand, and varied ratio of alkaline activator, which is described in Table 2 and 3. The mixture after preparation was immediately transferred to determine workability, then moulded and placed in a moist air room for 24 h. The mortar specimens were then demoulded and cured at room temperature (20±2°C) under a moisture controlled system. In addition, the mortar prisms for the corrosion test were immersed in water at room temperature (20±2°C) for 2 weeks before testing. That is to say, the development of alkaline activation showed less alteration after 14 days, and thus could be an optimum curing period for this observation. At the end of the curing period, the specimens were immersed in H_2SO_4 solution with pH 0.5, and monitored for the deterioration for 11 weeks.

RESULTS & DISCUSSION

Accelerated carbonation test

In this part of the study, a series of AAC (K0.6 series) exposed to CO_2 concentration of 3 vol% for 9 days, was observed. The cement paste specimens of AAH, AAB, AAMK and hybrids were subjected to accelerated carbonation testing after curing for 4 weeks at room temperature (20±2°C) under a moisture controlled system. The effect of the carbonation process on the AAC was accomplished by the Fourier Transform Infrared Spectrometer (FTIR) technique. The FTIR spectra were compared between the specimens before and after accelerated carbonation testing as shown in Figure 1.

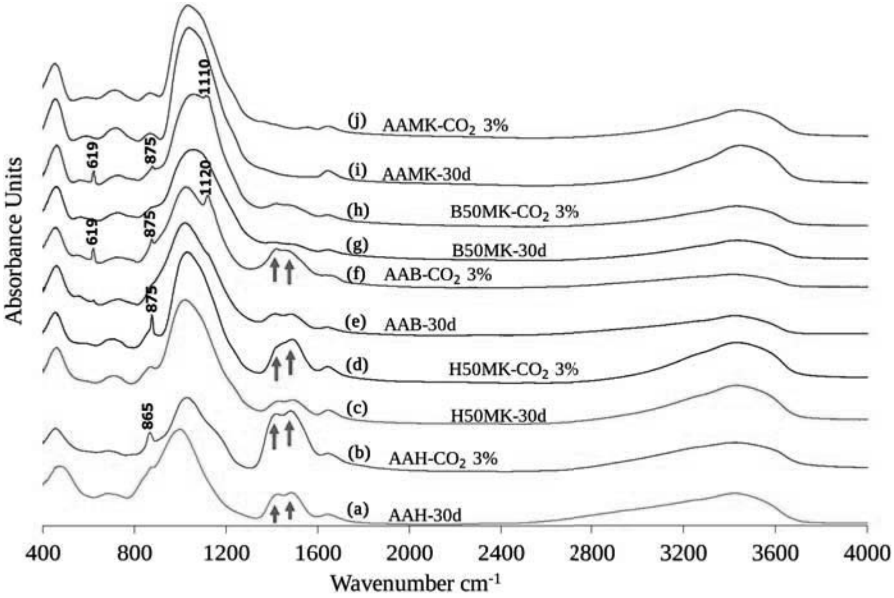

Figure 1. Comparison between FTIR spectra of AAC (K0.6 series) before and after the accelerated carbonation test, exposed to CO_2 concentration of 3 vol% for 9 days.

The mid-infrared spectrum of vaterite was measured by [11]. The presence of vaterite was indicated by the asymmetric stretch, v_3, which is split off as obviously seen in the bands at about 1420 cm^{-1} and 1490 cm^{-1} in Figure 1(a) (before exposure). The further reaction is detected by increasing of the intensity of the vaterite phase in Figure 1(b) after exposed to CO_2. A similar effect was also detected in Figures 1(d) and 1(f), while only small intensity could be detected in Figure 1(h). An intense band in the 1450 cm^{-1} region, is also reported as characteristic of alkaline carbonates [12], where the lowering of pH due to system carbonation reduced the ash activation rate but did not interrupt it altogether. The finding also demonstrates that this effect corresponded to CaO content of starting materials used as a comparison between AAH and AAB. These kinds of carbonation reactions did not appear in Figures 1(i) and 1(j) of AAMK series. The results are further evidence by FTIR, as given in Figures 1(a) to 1(h). The vibrational bands at about 875 cm^{-1} could be attributed to the modes of calcite formation [13], which was noticed as the appearance a weak shoulder in the series of specimens before carbonation. Moreover, Figures 1(e) to 1(h) of AAB and AAMK/B series indicated the characteristic bands of CO_3^{2-} cancrinite formation, which consisted of the characteristic fingerprint peak occurring at approximately 619 cm^{-1} (bending vibrations of SiO_4^{2-} ions and tetrahedral framework), and the asymmetric stretch for SiO_4^{2-} stretching vibrations of the tetrahedral framework at about 1110 and 1120 cm^{-1} [14-16]. These findings were confirmed by the previous work [12], indicating that sodium bicarbonate formed in a number of AAB series.

As a consequence of CaO content from the starting materials, a diversity of carbonate phase formations, i.e., vaterite, calcite, and cancrinite, was detected. This finding was similar to the effect observed in the previous studies. Bakharev et al. [17] found that the resistance of AAH concrete to carbonation was lower than that of OPC concrete, and AAH concrete had higher strength loss and depth of carbonation than AAH concrete in both tests, i.e., exposure to carbonated solution and atmosphere containing high CO_2. In addition, the hybrid binder of AAH-OPC revealed that the carbonation of Calcium Silicate Hydrate (C–S–H) was extensive [18].

Corrosion testing in sulfuric acid (pH 0.5)

The corrosion of concrete structures in sewage environments is a major problem in most areas around the world. The deterioration process is, namely, Microbiologically Induced Corrosion (MIC), which also known as H_2S sewer corrosion. The microbes oxidise the sulfur present in H_2S to H_2SO_4 which causes corrosion of sewer crowns, especially, in areas of extensive corrosion having

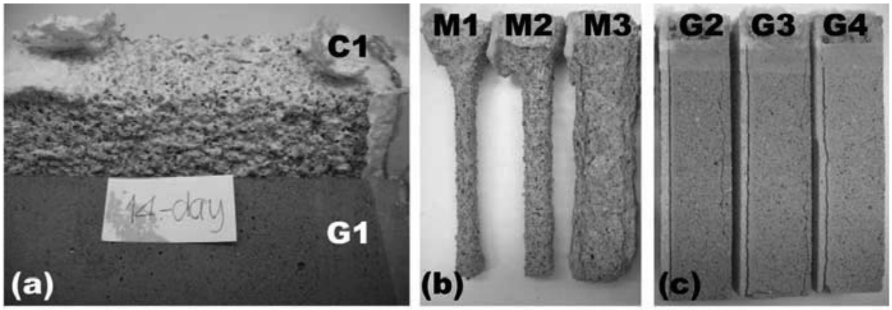

Figure 2. A comparison between OPC and AAC mortars immersed in H_2SO_4 with pH 0.5:
(a) OPC and AAC mortars at 2 weeks, (b) Modified-OPC mortars with AAC coating layer at 11 weeks, and (c) AAC mortars with AAC coating layer at 11 weeks.

low pH values of 1 to 2 [19]. In order to prevent this kind of corrosion, high-performance corrosion resistant material is still in needed for improvement to reach a satisfactory level. A possible solution for this problem could be achieved through a new type of AAC binders.

This part of the study, AAC mortars containing slag (H), brown-coal fly ash (B), and metakaolin (MK), were synthesized with K-based alkaline activator K0.5. After 14 days of immersion (Figure 2(a)), the AAC mortar (G1) revealed much better corrosion resistance against sulfuric acid compared to OPC mortar (C1), though C1 had a much longer curing period for 9 months. The reduction of w/c ratio from 0.5 to 0.35 as well as the OPC content in binder, showed better performance of modified-OPC (M1, M2, and M3). The decrease of OPC content of M3 resulted in a slower corrosion rate (Figure 2(b)). However, the AAC mortars (G2, G3, and G4) obviously demonstrated superior performance against corrosion in sulfuric acid compared to the OPC-based mortars (Figure 2(c)). In the results of corrosion studies between AAC and modified OPC mortars against the H_2SO_4 attack under the same conditions, the AAC mortar possessed potential for acid resistance compared to that of the conventional cement-based mortar. Therefore, a series of AAMK, AAB, and AAH with systematic MK replacement were taken for further observation.

In the following series of corrosion observations (shown in Figures 3 to 6), the AAC mortar specimens were prepared in the same mixture proportions as used for mechanical strength investigation published last year [20]. Whereas both flexural and compressive strengths have proven a tendency to increase with increasing time over 5 years. The specimens were preserved in a closed container under room temperature for 7 weeks before corrosion testing. During the test period of 9 weeks, the specimens were immersed only a half in H_2SO_4 with pH 0.5, and stored in the closed container under room temperature control. The H_2SO_4 acid solution was changed once every two days to control pH level. At the specified period of testing time, specimens were taken to observe. Each Figure represents a comparison between 2 types of alkaline activator (K0.5 and K0.64) with that of the same source of starting materials. Figure 3(a) shows the degradation of AAMK and AAB mortars after being immersed in H_2SO_4 acid for 2 days. A comparison between 2 types of alkaline activator revealed that the higher content of alkali (K0.64) was a factor influencing deterioration.

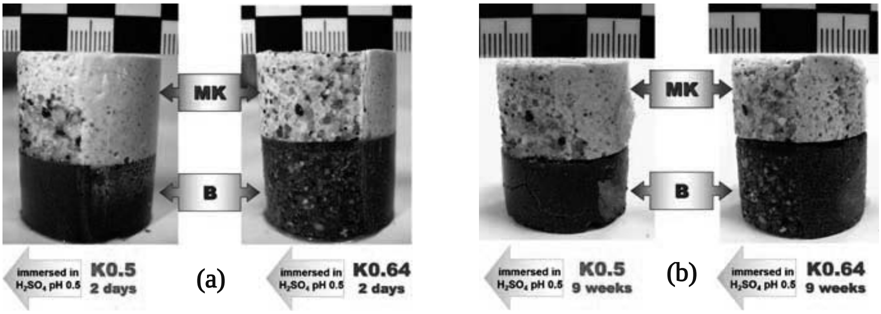

Figure 3. AAMK and AAFA mortars (K0.5 and K0.64) immersed in H_2SO_4 with pH 0.5:
(a) after 2 days, (b) after 9 weeks.

With increasing immersion time (Figure 3(b)), the deterioration exhibited less change for these two types of AAC mortars. As compared between sources of starting materials, AAB mortar tended to resist acid attack better than did AAMK mortar. However, the AAB mortar (K0.5) experienced cracks as a result of expansion in microstructure.

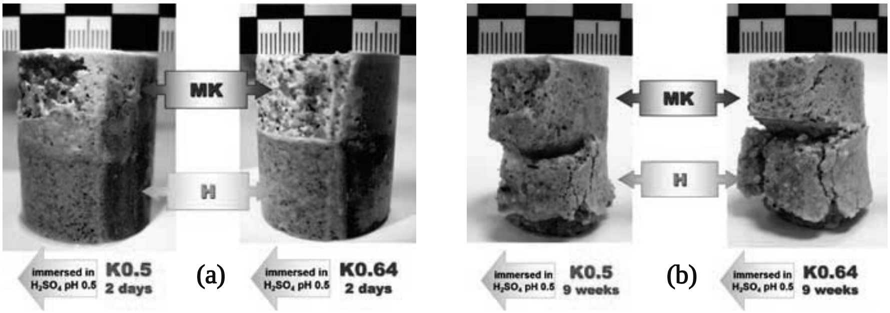

Figure 4. AAMK and AAH mortars (K0.5 and K0.64) immersed in H_2SO_4 with pH 0.5:
(a) after 2 days, (b) after 9 weeks.

The corrosion reaction of AAH mortars in Figure 4(a) and 4(b) was far different compared to AAMK and AAB mortars as shown in Figure 3. The degradation of AAH mortars had an even greater detrimental effect as a consequence of CaO content, which resulted expansion of mortar specimens. Between the two types of alkaline activator, it was difficult to compare the difference in long-term specimens.

Degradation effects with particular consideration of MK replacement in AAB and AAH series are presented in Figures 5 and 6, respectively. For the AAB series, the influence of MK replacement on degradation effect cannot be seen clearly compared to the role of alkaline content (K0.5 and K0.64), which exhibited the similar effects as found in AAMK and AAB mortars. Contrary to the AAH series in Figure 6, CaO content in slag played an important role in the corrosion mechanism, and led to total damage in pure slag mortar. The degradation effect significantly decreased with increasing MK replacement. That is to say, the corrosion resistance increased with decreasing CaO content. The results were not satisfactory for durability performance, however, they occurred exactly as expected.

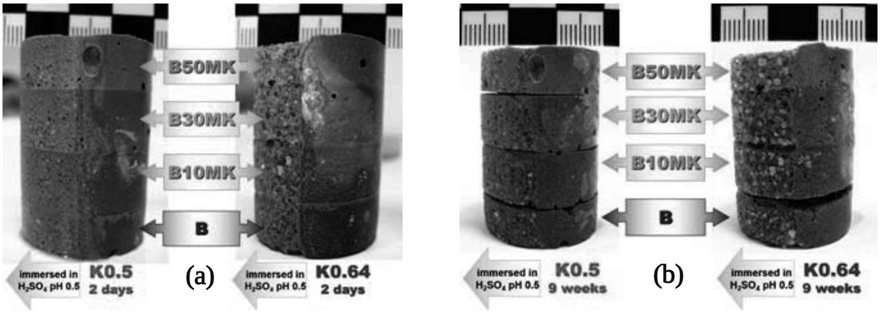

Figure 5. AAB and AAB/MK mortars (K0.5 and K0.64) immersed in H_2SO_4 with pH 0.5:
(a) after 2 days, (b) after 9 weeks.

The results of the investigation reported by ref. [21] show that AAH concrete had a high resistance in acetic acid solution of pH = 4, superior to the durability of OPC concrete. Contrary to the results of the investigation in sulfuric acid solution [22], the introduction of KOH in AAB samples caused an increase in the average pore diameter and reduced its durability. This could be explained by the depolymerization of aluminosilicate polymers and liberation of silicic acid, replacement of Na and K cations by hydrogen or hydronium ions, and dealumination of the aluminosilicate structure. It was also connected to condensation of siliceous polymers and zeolites, which in some cases led to a significant loss of strength.

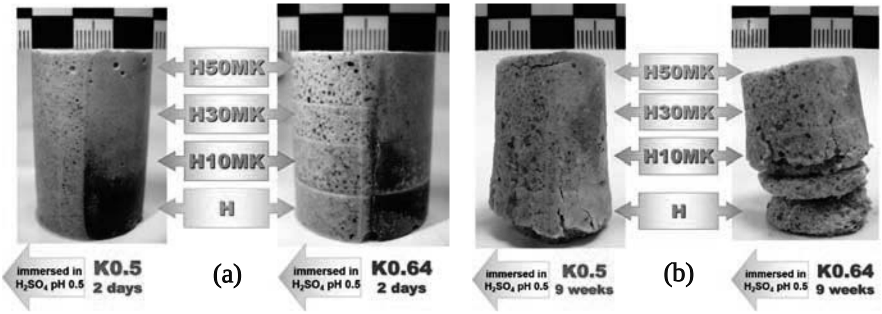

Figure 6. AAH and AAH/MK mortars (K0.5 and K0.64) immersed in H_2SO_4 with pH 0.5:
(a) after 2 days, (b) after 9 weeks.

SUMMARY AND CONCLUSION

The identification and observation in this study were focused mainly on application in the cement and concrete industry. These results are very encouraging and fulfilled the objectives of this study. Moreover, the observations tended to confirm the findings of several previous studies on durability performance of AAC binders.

One of the most important findings from this study was that the relationship between flexural and compressive strengths of AAC mortars could be characterized into two trends based on well-known empirical formulae, i.e., the Alkaline-alkaline-earth binding system and the Alkaline binding system or polysialate network [20]. This relationship can identify and classify the chemical components of starting materials into fundamental engineering properties, i.e., flexural and compressive strengths. This finding has been proven to be true from the data gathered in this study. Investigations were carried out using different starting materials and mixture proportions, and also under various concentrations of alkali activators.

According to the observations on durability performance, the AAC binders provide a superior solution for the application under aggressive environments compared to OPC binders. Despite the fact that the CaO content in starting materials and concentration of alkaline solution play an important role in the durability aspects of this material, these parameters are under control and reveal potential for further development.

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