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REVIEW

Alkali-activated materials: advances on accelerated and long-term durability assessment and methodologies—a short review

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Abstract
Developments in material testing have brought about the invention of some durability assessment methodologies for alkali-activated materials. This study reported advances in accelerated and long-term durability assessment and methodologies for alkali-activated composites (AAC). For both alkali-activated materials (AAM) and ordinary Portland cement (OPC)-based composites, the common methods such as increased acid concentration, standard non-accelerated test, wetting and drying cycling, brushing were assessed. The study assessed common methods: increased acid concentration, standard non-accelerated test, wetting and drying cycling, and brushing. The discussion also identified the limitations associated with the accelerated and long-term durability assessment in AAM composite. Some limitations include concrete pore solution concentration, precursor type, and admixture. In AAMs, pore size is within ranges > 1 µm and < 20 nm, which is an indication of insignificant porosity. Also, the compressive strength coefficient of AAM mortars was better than the cement mortar after 75 cycles. Finally, the study revealed the most appropriate mechanism for measuring the durability of AAM composite, which could be well utilized in the construction field.

Keywords Durability · Alkali-activated material · Pore solution · Degradation · Chemical reaction · Hydration

Introduction

In the construction industry of today's world, ordinary Portland cement (OPC) concrete has been the most widely used due to its versatility, cost, and outstanding properties [1]. However, durability concern has been one of the most critical factors for construction materials. It is dependent primarily on the material's useful life in actual service conditions, such as its exposure to aggressive medium [2, 3]. In a true sense, durability cannot be regarded as an intrinsic attribute of any material. Its severity lies in in-service conditions, which vary based on the type of exposure and duration the concrete is exposed. Concrete corrosion is a major end process of concrete exposed to a severe environment. Corrosion of reinforced concrete leads to delamination and blemishing, cracking, and in the worst case, structural failure. Corrosion initiates in a cementitious composite due to the exposure of the matrix to carbon dioxide (CO_2) and chloride ingress [3, 4]. Due to concrete making contact with the substances mentioned above, structural damage has become imminent, thus causing outrageous building repair and maintenance costs. The sources of chloride ions ingress in concrete include marine bodies, chemical contamination of concrete, using de-icing salts, and the use of chloride-rich additives during concrete production. Corrosion is more critical around the embedded steel in concrete. From there, it gets to a critical concentration, thereby resulting in the pitting corrosion of the steel after damaging the passive layer [5].

Despite the numerous benefits of concrete, extensive studies over the decades have indicated that the production of OPC concrete accounts for a significant portion of the energy consumption in the construction industry. This energy consumption has been estimated to be about 40% of the world's total energy consumption [6, 7]. On the other hand, alkali-activated materials (AAMs) can be used as a

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sustainable alternative to OPC materials as OPC is totally eliminated. AAMs are produced using a binder synthesized from an activator and aluminosilicate precursors to form a gel that hardens similarly to that of OPC [8–11]. Thus, AAMs are deemed to have low embodied energy and low embodied carbon. In addition, the precursors used in AAMs are mostly waste materials/by-products such as fly ash, slag, and silica fume [12–15]. Locally available materials such as laterite can also be used in the production of AAMs [16–18]. The use of these locally available materials and waste materials/by-products to replace OPC would also result in a significant reduction in the cost of construction materials and effective management of various waste materials. More improvement in the sustainability of AAMs can be achieved with the use of alternative activators compared to the conventional ones which are sodium hydroxide and sodium silicate [19–24]. Various waste and recycled materials can also be incorporated into AAMs as aggregate [25, 26].

Extensive studies over the years have shown that AAMs can exhibit similar or higher performance compared to that of OPC materials [27–29]. For example, AAMs made with slag as precursor have been found to demonstrate high durability to freeze–thawing, acid, and sulphate attacks owing to the absence of Portlandite and the presence of lower Ca/Si ratios in the C-S–H gel in its matrix structure (microstructure). OPC is deficient in the aforementioned microstructural features [4, 5, 8], making it susceptible to chemical attacks. However, low fracture toughness with quasi-brittle behaviour is evident in certain types of AAMs [30, 31]. However, compared to OPC materials, there is limited knowledge on accelerated tests and the long-term durability performance of AAMs [32].

AAMs are mostly assessed using the conventional methods available for OPC materials. However, the use of these methods for AAMs could be ineffective as the composition of AAMs differs from that of OPC materials. As the degradation process of construction materials such as OPC occurs with time, various accelerated tests have been devised to evaluate the long-term durability of the performance within a short period of time. However, there is limited availability of these accelerated tests to evaluate the long-term performance of AAMs. Thus, this paper aims to provide a comprehensive overview of the advances in the durability assessment of AAMs. Special attention is paid to methodologies and procedures of durability tests involving AAM composites. While not all alkali-activated materials systems are covered in this study, leading industrial products used as AAMs are considered. A lower water requirement and suitable rheological properties are the new generation AMMs systems[33]. Durability issues emanate in the composites due to chloride and carbon dioxide infiltration around the embedded reinforcement bars, freeze–thawing and sulphate attacks on pastes, and finally, efflorescence and acid resistance issues. Thus, this study renders detailed durability performances of AAMS and special consideration to testing methodologies and reveals the potential challenges of the processes involved.

Methodology

This study focuses various durability issues in alkali-activated materials and drawing comparisons among the various methods of assessing the durability of the materials, in terms of accelerated and long-term durability techniques. The study also explored several durability assessment techniques that may be applied to AAMs, and the study also emphasizes the resistance of AAM in aggressive medium and compared to the OPC-based composites. Figure 1 presents the various stages on this study.

Several features are measured in concrete or cementitious materials to ascertain their durability. AAMs' most applicable durability properties are porosity and permeability, chloride, carbonation and corrosion, sulphate and acid attacks, efflorescence, and fire resistance assessment.

Permeability and porosity

The deleterious substances (sulphates and chlorides), through mass transport mechanisms, are distributed in capillary pores in the AAM composite[34]. Reports indicate that pore size in AAM composite is within ranges > 1 µm and < 20 nm, thus implying that insignificant porosity occurs in the size range. Notwithstanding, this range is small compared to that of a similar grade of OPC (unimodal pore size distribution of 10–100 nm). A mercury intrusion porosimetry (MIP) device is used to assess porosity in composites. However, there is limited information about the use of MIP for AAM composite. Thus, it is clear that variation in the pore size distribution of AAMs relative to OPC concrete has a somewhat significant influence on the durability properties

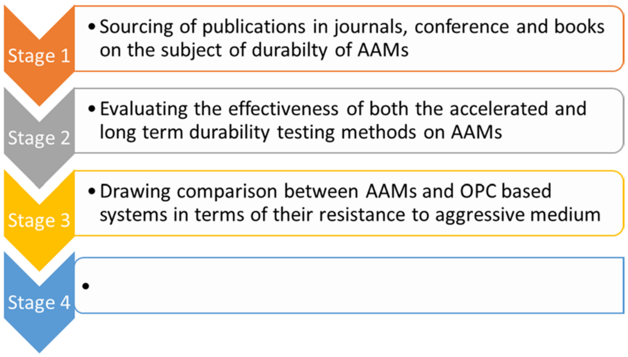

Fig. 1 Flow chart of the stages of this research

of AAMs compared to the former [1]. The activation process and activator type also contribute to the pore distribution in AAMs. For instance, AAMs activated with potassium exhibit a minimal pore diameter than sodium-based activators. Provis and Deventer[35] show that sorptivity features in AAMs are comparable in range to the OPC-based composites. In practice, lower mixing water and silica-rich activator aid reduction in capillary sorptivity AAMs. The aggregate-paste zone in the AAM composite is less porous and stronger than the normal concrete. The main backing to the better pore structure in AAMs is traceable to the chemical interaction between alkali binder and aggregate particles.

Chloride, carbonation, and corrosion

Chloride ingress in AAM is not well pronounced like in normal concrete, mainly due to pore's efficient pore structures and fluidity. More reliable performance of cementitious composite in terms of resistance to chloride ingress can be measured using older Nordtest [36, 37] and BS EN 12,390–11 [38]. The test methods utilize powder samples for determining soluble (water and acid) chlorides. The actions of pore fluid in AAM composite require more in-depth investigation into the chemistry of the materials. Studies [39–41] have shown that AAM composite demonstrates higher chloride resistance potential than the same grade of OPC concrete.

The pore fluid in AAM is affected by the nature and type of precursors and activators. The former also influences the electrolyte for the steel corrosion process and protection. While in the normal concrete, chloride concentration at threshold levels is seen at the steel interface, which also initiates the corrosion process, the AAM composite is different. The products of hydration for the precursor utilized and limited calcium hydroxide $Ca(OH)_2$ buffers delay corrosion of steel in AAM composites. The differences in the resistance to chloride attack and initiation of corrosion in normal concrete and AAM composite render the accelerated durability tests method and the service life prediction models of conventional concrete unsuitable for AAM concrete.

In terms of carbonation resistance, AAM containing blast furnace slag or a blend with metakaolin has exhibited a higher carbonation rate than the normal concrete, owing to insufficient $Ca(OH)_2$ as a hydration product. Similarly, the study by Puertas et al. [42] and Bakharev et al. [43] where sodium hydroxide and sodium silicate were used to activate slag indicated that such AAMs undergo faster carbonation compared to that of OPC materials. The rapid degradation of these AAMs as a result of the faster carbonation is detrimental to both the mechanical and durability performance of the materials [44, 45]. However, the faster degradation of AAMs due to carbonation could result from the accelerated test used, which does not represent the in-service conditions. Thus, it is imminent and critical to devise tests that can be used to assess the long-term durability of AAMs accurately. Nevertheless, the carbonation resistance of AAMs is greatly influenced by the type of precursor and activator used. A comprehensive study carried out by Bernal et al. [46] indicated that the incorporation of metakaolin as a precursor in the synthesis of AAMs would increase its vulnerability to carbonation. The method used to evaluate the carbonation resistance of the AAMs is an acceleration test that involves exposing the AAM samples to carbon dioxide concentrations of 3% at a relative humidity and temperature of 65% and 2 °C, respectively. In the same study, it was found that the carbonation rate increased with lower silica modulus for AAMs made with solely sole slag as the precursor.

Meanwhile, carbonation can be mitigated by selecting an appropriate mix design, type, and activator concentration. Moreover, it is also opined that higher binder concentration could improve carbonation resistance in AAM. However, future studies may need to explore why the accelerated carbonation tests in the AAM composite contradict that of the performance of OPC concrete and vary from the long-term exposure of the composite.

It should be noted that the standard phenolphthalein carbonation test at a rate of under 1 mm/year using RILEM TC 224[35] is unsuitable for AAM composite, as this was designed only for OPC concrete based on its chemistry and hydration of $Ca(OH)_2$. Thus, there is a need for more in-depth investigations and probably the use of other novel alternative methods to apply the test to AAM composites. In addition, there is also the effect of carbonation shrinkage in the cementitious composite. The concept of carbonation shrinkage in new materials such as AAM based has not been adequately reported in the literature, and this needs in-depth studies. It is currently not clear what is the accelerated correlation data with the natural condition. This issue has persisted because of the variation of the process of diffusing CO_2 and pore fluid chemical composition to OPC with a greater concentration of $Ca(OH)_2$. The concentration of CO2 in the environment affects the carbonation rate to a large extent in AAM composite, and according to researchers [35, 47, 48], both the pore fluid carbonation and gel degradation rates are modified at a higher CO2 exposure of composite.

Efflorescence

One of the issues with certain types of AAMs is efflorescence [49]. Three main factors: high pore fluid alkali concentration, AAM porous microstructure, and Na weak binding of sodium (Na) in the aluminosilicate gel structure contribute to efflorescence in AAM composites [50, 51]. However, the rate or extent of efflorescence is influenced by the type of alkali metal, reaction conditions, and reactivity of the precursors [52, 53]. Thus, excess sodium oxide not involved in the AAM synthesis could result in efflorescence [54]. Efflorescence can be seen as a whitish material formed on the surfaces of AAMs, which is formed due to the reaction between the excess sodium oxide and atmospheric carbon dioxide. The whitish material has been reported to be composed of hydrous alkali carbonate ($Na_2CO_3.7H_2O$) [52, 54]. However, the amount of structural H_2O formed in the alkali carbonate is dependent on the humidity and temperature and the AAMs are subjected to [55]. The mechanism involved in the formation of efflorescence can be summarized as shown in Eqs. 1 and 2 [56]. In contrast to the carbonation method discussed earlier, the products formed due to efflorescence are visible to the human eyes and may not deteriorate the AAM performance but may alter its aesthetic features [54].

$$CO_{2(g)} + 20H^{-}_{(aq)} \rightarrow CO^{2-}_{3(aq)} + H_2O \tag{1}$$

$$2Na^{+}_{(aq)} + CO^{2-}_{3(aq)} + 7H_2O \rightarrow Na_2CO_3.7H_2O_s \tag{2}$$

Efflorescence in AAMs can be reduced or eliminated using various methods such as use of alternative activator to replace sodium hydroxide. Several studies have utilized potassium hydroxide as an alternative to sodium hydroxide in order to reduce efflorescence in AAMs [54, 57]. The study by Najafi Kani et al. [58] also indicated that the alkali leaching in AAMs can be correlated with the occurrence of efflorescence. The study found out that for AAMs with lower amount of alkali leaching, there was lower amount of efflorescence occurrence. It was also recommended that efflorescence in AAMs can be reduced with the use of hydrothermal curing or the incorporation of admixtures rich in alumina. The presence of additional alumina in the AAMs due to the incorporation of admixtures would reduce alkali mobility and enhance crosslinking [58]. Slag which is a precursor can also be incorporated into fly ash-based AAMs to reduce efflorescence. However, the mechanism in which slag can reduce efflorescence is more related to its ability to reduce the permeability of the AAMs, thereby inhibiting alkali leaching [58].

Currently, there exists no explicit test method for evaluating the efflorescence of AAMs. Most assessments are carried out using visual assessment or ASTM D7072 or the efflorescence assessment of latex-coated substrates [59, 60]. Optical microscopic image of efflorescence formation on AAMs is presented in Fig. 2, while Fig. 3 shows formation of efflorescence on some AAMs captured by a digital camera [56]. The study by Weng et al. [60] also indicated that image analysis can be used to assess efflorescence. Figure 4 shows the use of image analysis in the quantification of efflorescence amount.

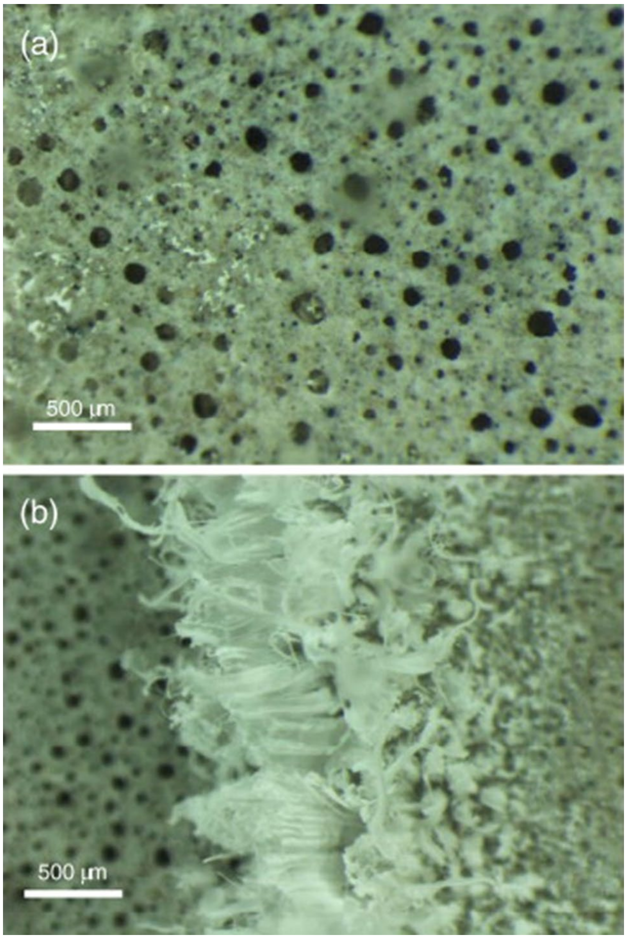

Fig. 2 Optical micrograph showing formation of efflorescence on AAMs with time **a** 24 h **b** 7 days [56]

Freeze–thaw resistance

In OPC concrete, the physical properties of materials like matrix strength, pore saturation, voids, and pore structure affect the freeze–thaw resistance of the concrete. In the case of OPC concrete, there is no significant contribution of hydration product composition to durability. Although it has not been established by research, the factors mentioned above will also affect freeze–thaw resistance in AAM composites. In blast furnace slag-based AAM, owing to its high ionic concentration, pore fluid in the matrix can freeze at a temperature below – 50 °C. Thus, it effectively improves freeze–thaw resistance. Moreover, AAM composites are also known to have appreciable resistance to frost and frost-salt attacks. This is also the case for the in-service state of the AAM structures, which is far better than the same grade of

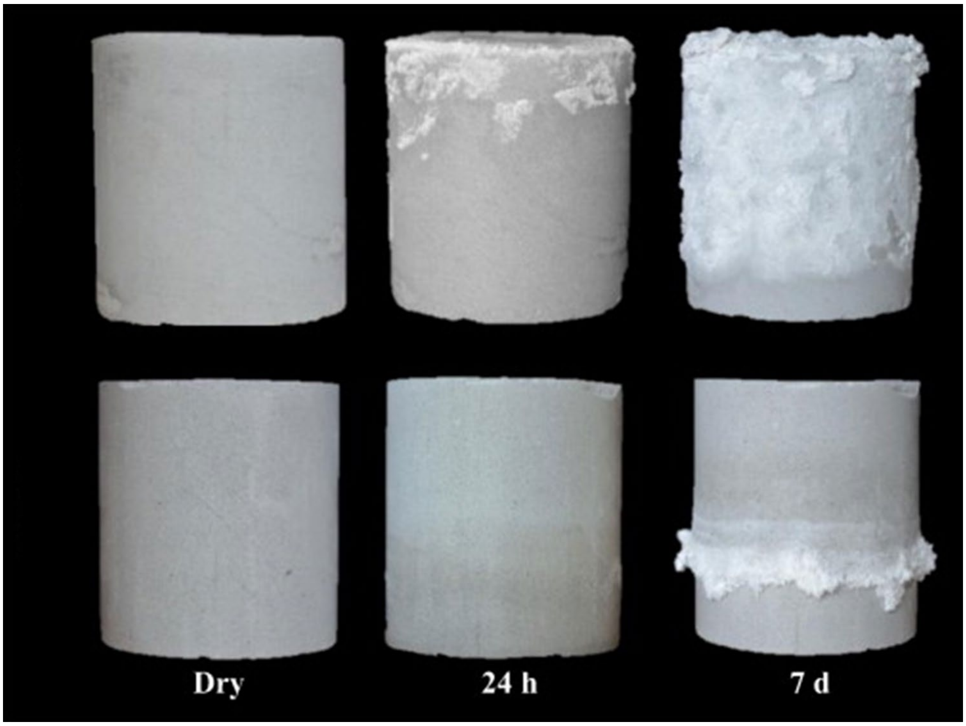

Fig. 3 Digital image showing formation of efflorescence on some AAMs [56]

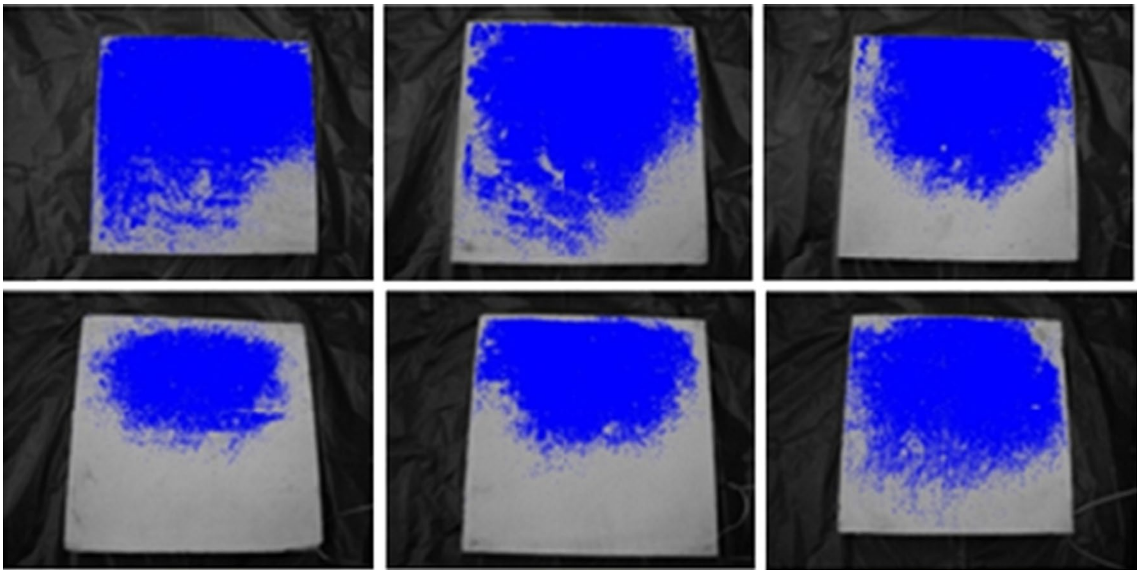

Fig. 4 Use of image analysis in the detection and quantification of efflorescence [60]

OPC concrete [61]. AAM composite is also not severely affected by freezing and thawing early age.

In contrast, a report showed that AAM composite demonstrated the same poor mix of OPC concrete [62]. According to the authors, AAM composite lacks Portlandite and ettringite minerals which should control the higher free water in the matrix pores. With the presence of the crystal phases, freeing of pore-free waters will be prevented. Freeze–thaw techniques as provided by standards should be appropriate for testing AAM composite. However, further attention may be paid to procuring and presaturation requirements of specimens.

Sulphate and acid resistance

The chemical actions of hydration products in OPC concrete can make it susceptible to sulphate attack [64]. Eventually, there is the disintegration of concrete by expansive ettringite or the formation of gypsum. However, this is not the case with low-calcium-based AAM, which exhibits better resistance to sulphate solution. In addition, using precursors like fly ash, a low calcium material reduces hydration products in AAM composite, making it free from expansive ettringite C-S–H that could cause damage [65]. Thus, limitations in hydration products ettringite formation make AAM composite somewhat superior to OPC concrete in acid resistance.

The experimental findings of fly ash-based AAM composite resistance to sulphate attack are presented in Fig. 5. Variation of ion concentration and pH has been presented. The dissolution of AAM silica-rich content or unreacted activator is considered to have yielded the Si discovered in the sulphate solution. There was an increase in Ca concentration, which implies a poor sulphate attack resistance of calcium-rich hydrates (C-(A)-S–H gel and C-S–H gel)than the N-A-S–H gel. A related study indicated that the Ca dissolution could be attributed to ion exchange or reaction with Na2SO4 [63]. The studies above also agree with Zheng et al. [64], who revealed that the resistance of AAM mortars to sodium sulphate is better than OPC mortar under dry–wet cycling. The compressive strength coefficient of AAM mortars was better than the cement mortar after 75 cycles.

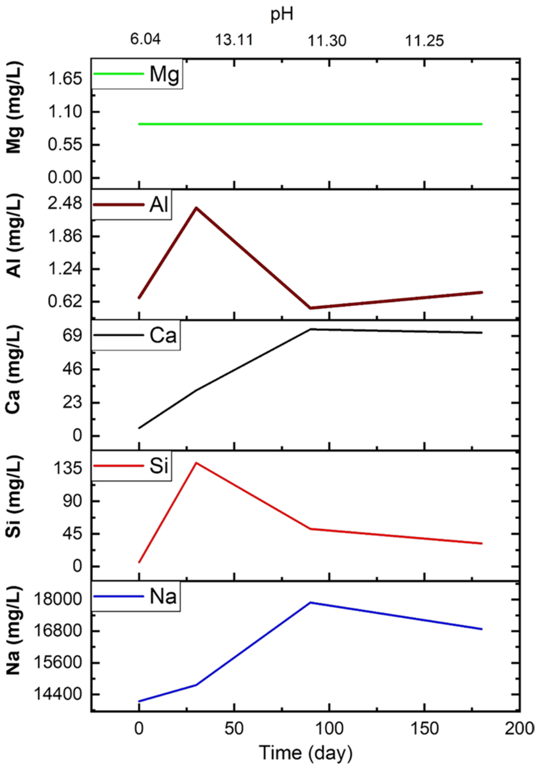

Fig. 5 pH and ion concentration of fly ash-based AAM in 5% Na2SO4 solution (adapted from Džunuzović et al. [65][65])

Fire resistance

RILEM reports [35] have shown that AAM composites possess high-temperature resistance due to low-calcium content.. In the structure, the internal pressure build-up is prevented via amorphous gel, thus enhancing the fire resistance. In this case, spalling or splitting of the concrete, which is the norm in OPC concrete, is prevented.

Fireplug and furnace tests for AAM composites have produced good insulation services [36]. There is variation in the fire resistance of AAM composite, depending on the type of precursor. For instance, fly ash-based AAM produces better fire resistance than a metakaolin precursor, as reported by researchers [12]. The researchers attributed such variation of pore distribution to be responsible for permeating steams from the structures. However, the fire resistance of the metakaolin-based AAM may be enhanced by blending metakaolin with blast furnace slag [35]].

Alkali–silica reaction

Like cancer in humans, alkali–silica reaction (ASR) involves overtime deleterious swelling in concrete caused by reaction of reactive amorphous silica in aggregates and the highly alkaline cement paste. It should be noted that the amorphous silica is non-crystalline.

As a result of the chemical reaction, aggregates expand, and thus leading to the development of soluble and viscous gel of sodium silicate ($Na_2SiO_3 \cdot n\ H_2O$). Subsequently, there is an increase in volume due to the swelling of the hygroscopic gel when absorbing water.

Thus, pressure is exerted in the siliceous aggregate due to the expansion, resulting in the spalling and eventually strength loss in concrete. Moreover, due to ASR, concrete cracking can occur and lead to critical structural failures.

The ASR-induced expansion has been compared in OPC and alkali-activated material [69]. In OPC, the alkalis content (Na2Oeq% < 0.6%) is limited, which helps mitigate the ASR in the OPC mixture. The typical mechanism for cracking induced by ASR is illustrated in Fig. 6. Three stages are involved.

In Fig. 6, it is shown how the dissolution of poorly crystallized silica in aggregates occurs, the generation of silica ions following the continuous attack by the silanol groups, and finally, there is the precipitation and polycondensation of silica ions by the cations in concrete's pore solution.

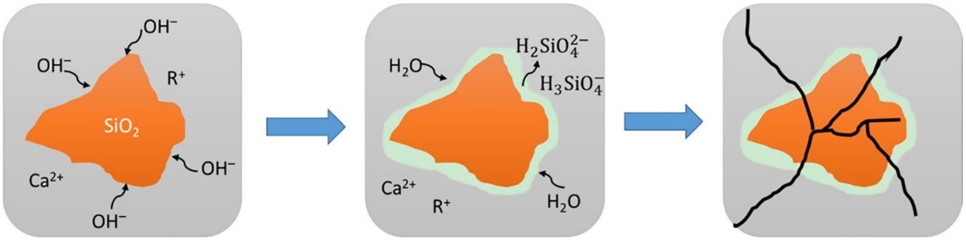

Fig. 6 The typical mechanism for induced cracking in ASR system [70]

Accelerated and long-term durability assessment and methodologies

With the advancements seen in research activities over the years, several innovative methods have been utilized to assess the durability properties of AAM and OPC composites. In Table 1, several test methods and details have been presented. Strategies adopted include wet and drying cycling, brushing, in situ, and continuous immersion. Also, the duration of testing varied from hours to those extended to several weeks.

Summary and future perspective

The focus of the current study is on critiquing the various durability issues in alkali-activated materials and more so comparing the various methods of assessing the durability of the materials, in terms of accelerated and long-term durability procedures. AAMs have been continuously developed with innovative materials such as slag, fly ash and metakaolin, but, durability phenomenon in AAM has not been overly explored. The study has pointed out several durability assessment techniques and the significant resistance of AAM in aggressive medium than the OPC-based composites. For the future research, there is a need to develop advanced characterization methods for reaction products or precursors—databases are required that provide the accurate description and thermodynamic prediction processes during the formation of gel in most low-calcium AAM composite. Also, the transport mechanism influences the durability performance of composite; hence, there is a need framework for understanding the transport mechanism in an AAM-based mixture. The future studies should also dwell on development of sustainable and durable composites using AAM in 3-D printed concrete, many more.

Conclusions

This study reported advances in accelerated and long-term durability assessment and methodologies for alkali-activated composites. The following conclusions were drawn from the study:

(i) Reports from several investigations generally showed that AAMs have satisfactory durability performances in aggressive mediums, such as sulphate, acid, fire, and freeze and thawing. Moreover, the pore solution chemistry and microscale reactions and precursors influence the durability performance of AAMs.

(ii) On a general note, the well-known methods of testing long-term durability performances of AAMs (mostly for slag, fly ash, or their blends) are the same for OPC concrete. Although, inaccurate results may be obtained due to the chemistry and structure variations of the binders and OPC. Factors such as admixture dosage, pH, detection technique, and concrete pore solution affect the repeatability and reproducibility of accelerated carbonation and finding the critical chloride threshold level in CPT.

Table 1 Common durability assessment methodologies – OPC and AAM composites

Method	Specimen description	Water content/Curing	Solution concentration	Measure& intervals	Sources
Biogenic test, wetting anddrying cyclingBrushing methods.;This involves sulphuric acid samples rotation in acid solution at a hydrogen speed of 1 rev/h.,	Concrete samples made of blast furnace slag and high sulphateresistant Portlandcement. specimens of sizes: Ø200mm, 60mmØ200mm, 80mm20mm, 20 mm,50 mm	0.25, 0.36, 0.40, 0.43, 0.49	0 0.5% Hydrogen sulphide and concentration of 250 ppm achieved via sulphuric acid	Mass loss and change in length test duration is 5 days	[66]
Partial immersion of specimens in domesticsewage	100 mm, 70 mm, 70mmConcrete couponsProduced from already corroded slabs		Hydrogensulphide at a concentration of 0 ppm,5 ppm, 10 ppm,15 ppm, 25 ppmand 50 ppm	Surface pHSurface sulphateCorrosion rate0, 6, 12, 18, 24,33, and 45 weeks	[67]
Continuous immersion and in situ test specimens to be immersed in solution	Ø60mm, 140 mm;20 mm, 20 mm—20mmcalciumAluminate, and OPC concrete samples	0.37	In situ test:biogeniccorrosion;Laboratory test:sulphuric acidLaboratory test: pH 1and 2	replacing calcium andaluminium-concentrations inacidic solution. And then measuring the Mass loss of sample	[68]
Immersion of samples in solution, and Dry and dry cycling	70 mm, 70 mm, 280mmConcrete samples made of OPC, ground granulated blast furnace slag, and fly ash	0.35 Moist-curefor 28 daysat 20 ± 3 oC	5% Sodiumsulphatesolution	Measuring the pore size distribution and relative dynamicelastic modulusat 1, 2, 3, 4, 5, 7, 9,11, 15, 19, 20,25 weeks	[69]
Biogenic method, andin situ test done by placing mortarSamples in situ,	Mortar samples of 100 mm-thick coresample in pipe 40 mm—40 mm, 160 mm	0.65 Water-curefor 28 daysat 20 -22 oC	Hydrogensulphide solution at a concentration of 400 ppm	Measuring the corrosion depth using the biogenicchamber test at42, 72, 180 days	[70]
spraying andperiodic soaking of concrete samples for days of cycling and spraying the samples at 60 ml/h for 5 daysand dry for 1 day	Ordinary Portland cement Concrete samples of sizes Ø100mm, 200 mm;Ø75mm, 150 mm;Ø50mm, 100 mm	0.25, 0.30, 0.35, 0.40, 0.50 Moist-curing of samples for period of 28 days at 23 ± 1 oC	Sulphuric acid 0.3%, 0.6%, 1.0%,1.5%, 2.0%, 2.5%, 3.0%	Measuring the Mass loss and Corrosion depth of specimens at 1, 2, 3, 4, 6, 8,and 13 weeks	[71]
ContinuousImmersion of the samples inthe solution	Mortar samples of sizes Ø25mm, 25 mm;50 mm—50 mm, 50 mm	Placing the specimens in moulds for 24hoursat 23 ± 2 oC, and then leaving it over 98% relativehumidity,demoulding, and curing it underthis conditionup to 28 days	This requires simulating the possible service conditions, and then replacing thesolution following the period of inspection	Assessments by visual inspection, and measuring change inlength, mass, and compressiveStrength of samples at 1,7,14,28,56,and 84 days	[72]

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Author contribution PA: Conceptualization, writing—review and editing, NB, AA and OO: Writing – review and editing. All authors read and approved the final manuscript.

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Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

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