# EFFECT OF THE USE OF AAC IN MITIGATING THE ALKALI-AGGREGATE REACTION

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#### Abstract

Alkali-aggregate reaction is a pathology directly linked to interactions between solubilized alkali hydroxides in the pore structure concrete and other chemical species from aggregates. In the presence of excessive water, these chemical interactions lead to the precipitation of gel-like phases within aggregates, paste-aggregate interface and cement matrix, causing expansion due to internal pressure in concretes that can promote disruption. It appears that the volume expansion of silica gels tends to have a maximum at the total dissolved silica. Both alkali and silica are needed to chemically stabilise alkaline activated cements (AAC-geopolymers). Therefore, this study aims to evaluate the potential use of geopolymers as mitigating agent of AAR. Four formulations were carried out in the study, three metakaolin based AAC were synthesized with different alkali types (100% sodium silicate, 100% potassium silicate and 50% of sodium silicate and 50% potassium silicate). Portland cement mortar (CPV-ARI) was used as reference. Reactive aggregate (Pyrex) was used as aggregate in all mortars. All materials and samples were characterized by means of their chemical, mineralogical and microstructural properties. Prismatic mortars bars (25x25x285 mm) were cast and tested performed by the accelerated expansion method according to NBR 15577-4 and 15577-5. Scanning Electron Microscopy, Ultrasound analysis and X-ray Diffraction were used as complementary testing. Considerations are given with regards to the potential use of AAC as mitigating agent for AAR.

Keywords: AAC, Geopolymer, AAR, Mitigating Agent.

### 1 INTRODUCTION

It is known that concrete is currently one of the most widely used products on the planet. With the modernization of structures due to both architectural boldness as the verticalization of constructions, it is necessary structures with higher strength and, consequently, higher consumption of cement, increasing the reactivity of the concrete and in turn the possibility of the presenting of pathologies such as alkali-aggregate reaction. In addition, large amounts of CO<sub>2</sub>, a major greenhouse gases are released to the atmosphere during manufacture where cement is used high temperatures (1400-1500°C), making the process very expensive at energy levels.

The alkali–aggregate reaction is a chemical process that involves alkaline oxides generally deriving from the alkalis in the cement and certain forms of reactive silica present in the aggregate which may lead to dissolution of the aggregate and the formation of the AAR [1-3].

Some measures that has been successfully used to control expansion caused by AAR is the partial substitution of Portland cement with silica fume or fly ash [5-7]. The effect of the ash in curbing expansion is attributed to several factors, including the lower available calcium content in the system and reduced alkalinity in the solution saturating the concrete pores among many other possibilities. However, the presence of calcium has been still controversial and its role in the alkaliagregate reaction mechanisms is not fully understood. [8-10].

Studies show that in mortars and concretes the fly ash based alkali-activated, the Portland cement can be 100% replaced. These mortars and concretes, in general, are characterized by a high alkali content, but also by low calcium content [11-12]. This makes these materials exhibit a different behavior than Portland cement with respect to alkali-aggregate reaction.

In recent years, new cementitious materials began to be developed and tested. Most research with new materials aims to improve the mechanical, physical or chemical properties of binder

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materials, seeking increased energy efficiency, reduced  $CO_2$  release into the atmosphere, reducing pollution of water resources, among others, and the creation of high-performance materials that minimally degrade the environment [12-13].

Thus, the Alkali Activated Cement (AAC) are a class of inorganic polymeric materials with high mechanical strength, resistance to chemical attack and exposure to high temperatures, in addition to having good durability in comparison with Portland cement [12-27]. The generic term "geopolymer" used to describe the AAC was used for the first time in 1978 when a French researcher produced binders calcined based mixtures of kaolinite, limestone and dolomite, sodium hydroxide solutions at various concentrations, and subjected to temperature synthesis 150°C [28]. This material reported by Davidovits it is the same material belonging to the alkaline system Me<sub>2</sub>O-Me<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-H<sub>2</sub>O, discovered and described by Glukhovsky [29] and studied previously by Purdon [30].

Since the beginning of its research, AAC was thought of as an alternative material with the possibility of replacing Portland cement (OPC) and be used in traditional building technologies [31]. However, although currently there is considerable literature on the AAC related to its technology and applications in building [12, 24-27, 32-34], their studies are preferably based on some traditional precursors and traditional applications such as recovery of material with fire resistance, for immobilization of toxic and radioactive waste [12, 24, 31-34].

Thus, the aim of this study is assessing the potential of mortars AAC the metakaolinite basis as an agent mitigating the AAR and compare the systems similar (same content of water / cement ratio, the same formulation of the aggregate) materials alkali-activated cement with systems Portland cement base through procedures based on the described in ABNT NBR 15577-4 [35]. The experimental approach involved the use of a reactive aggregate (Pyrex) and AAC activated with three alkaline activators formulations (100% sodium silicate, 100% potassium silicate and a combination of 50% sodium silicate + 50% potassium silicate). The use of AAC as mitigating agent is justified by the conditions of the formation of AAR can promote the synthesis of the AAC, both the pH of the solution as the availability of alkalis, and require less energy to its structural organization compared the necessary energy the AAR. Besides the accelerated test expansion in bars were performed microanlytical analyzes as Scanning Electron Microscopy, Diffraction X-Rays and Fluorescence X-Rays, in order to identify differences between mortars material activated alkali-and Portland cement based.

### 2 MATERIALS AND METHODS

## 2.1 Materials

The materials used in this study were as follows:

• The metakaolinite used in the research was obtained from the calcination at 700°C for 2 hours kaolin supplied by Caulisa SA, kaolin processing industry located in the municipality of Juazeirinho, PB.

• Portland cement CPV-ARI from a Brazilian cement industry.

• The reactive aggregate used in this research was the pyrex, provided by an industry of Recife-PE. This aggregate is an extremely reactive amorphous material and rich in silica.

• As alkali activators used were sodium silicate supplied by Pernambuco Chemistry with S/A=2.17 (ratio  $SiO_2/Na_2O$ ) and potassium silicate supplied by Diatom with S/A=2.15 (ratio  $SiO_2/K_2O$ )

• Sodium hydroxide, supplied by VETEC with a purity of 97% was used to obtain the alkaline solution to be used in the accelerated test expansion, prepared in accordance with the recommended standard NBR 15577-4.

The aggregate was crushed, ground and sieved to the particle size distribution shown in Table 1. Table 2 gives the chemical characteristics of the raw materials used. The metakaolinite, cement and aggregate chemical composition was established by X-ray Fluorescence. The raw materials were also characterized for mineralogy and microstructure.

TABLE 1: The granulometry distribution of the aggregate used.

Sieve size		
Passing	Retained on	Mass, %
4,75 mm	2,36 mm	10
2,36 mm	1,18 mm	25
1,18 mm	600 µm	25
600 µm	300 µm	25
300 µm	150 µm	15

TABLE 2: Chemical composition of the raw materials.

Material	SiO <sub>2</sub>	$Al_2O_3$	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	TiO <sub>2</sub>	MgO	SO <sub>3</sub>	Others
Metakaolinite	62,96	34,61	1,34	0,63	0,07	0,05	0,03	0,12	0,00	0,19
Cement	20,91	1,94	0,84	2,42	0,51	63,94	0,26	3,62	5,18	0,38
Pyrex (Aggregate)	74,77	3,32	0,43	0,15	10,05	8,45	0,00	2,56	0,26	0,01

#### 2.2 Methods

Mortars preparation

The method used to evaluate the possible appearance of the ASR in the specimens was based on ABNT NBR 15577-4. This method estimates the deterioration that the ASR may potentially cause in cement mortars in 30 days.

The mortar specimens were made with the aggregates described above. Three series of specimens were prepared. The first series contained 100% sodium silicate, the second 100% potassium silicate and the third 50% sodium silicate + 50% potassium silicate. Prismatic specimens  $(25 \times 25 \times 285 \text{ mm})$  were made from the materials to be tested, Portland cement mortars (as a control) and AAC mortars base metakaolinite.

#### Mortars curing

OPC Mortars: The (deionized) water/cement ratio used in the Portland cement mortars was 0.47 (see Table 3). After an initial curing in a chamber at 23°C and 99% relative humidity for 24 h, the prisms were de-moulded, immersed in water and stored in sealed containers in an oven at 80°C for a further 24 h.

AAC Mortars: An alkaline solution (sodium silicate or potassium silicate or mix of sodium and potassium silicate) was used as the activator to make the AAC mortar, which was mixed in the same way as the Portland cement. The "solution/AAC" ratio was 0.47. After mixing, the specimens (in their respective moulds) were placed in an oven where the mortars were cured for 24h at 50°C in high humidity conditions. Finally, they were removed from their moulds and applied the same conditions as the OPC mortars.

#### TABLE 3: Mortar features.

		Binc	ler	Activa		
Mix	(1) Pyrex Content (g)	OPC Content (g)	Metakaolinite Content (g)	Sodium Silicate Content (g)	Potassium Silicate Content (g)	(2) L/S ratio
OPC	990	440	0	0	0	0,47
MK.SN	990	0	192	248	0	0,47
MK.SK	990	0	192	0	248	0,47
MK.SNK	900	0	192	124	124	0,47

(1)Reactive Aggregate, (2) water/cement ratio or solution/metakaolinite ratio in ACC systems.

#### Accelerated Test Expansion

After their respective curing processes, both the Portland cement and AAC mortar specimens were immersed in a 1 M NaOH solution and stored in sealed containers in an oven at 80°C. The specimens were routinely measured for length throughout the duration of the test to determine induced expansion. Finally, at the ages of 30 days, the specimens were studied and characterized by SEM/EDX, XRD, Ultrasound analysis and Compressive Strength Test.

#### Characterization

The samples were characterized mineralogically by XRD with the BRUKER D-2 Phaser diffractometer operating with Cu K $\alpha$  radiation, 30 kV and 10 mA, with scanning 20 between 5° and 65° with step 0.02°/s, microstructurally by SEM with FEI Quanta 450 microscope and Ultrasound analysis with PUNDIT PL-200 de 54 kHz.

### 3 RESULTS

### 3.1 XRD and SEM of Raw Materials

XRD analysis (Fig. 1(a)) showed that the aggregate (Pyrex) consisted primarily of quartz and tridimite, both source of SiO<sub>2</sub>. Unsurprisingly, the XRD analysis of the Portland cement revealed that the majority phase was alite (tricalcium silicate). The other phases normally present in Portland cement were also observed: belite, gypsum and the interstitial phase (aluminates and ferrites) (Fig. 1(b), OPC). The XRD analysis of the metakaolinite revealed that the majority phase was quartz and minority presence of mica (Fig. 1 (b), MK).

The Figure 2 shows the SEM micrograph of cement sample (a) and pyrex (b), where it can be seen, the cement fineness and pyrex geometry with well defined surface and angled corners.



FIGURE 1: (a) XRD patterns of aggregate (Pyrex), (b) XRD patterns of Portland cement (CP) and Metakaolinite (MK); Q quartz; Td trimidite; M mica; K kaolinite; Gy gypsum; C3S alite; C2S belite; C3A tricalcium aluminate; C4AF tetracalcium aluminoferrite; CaO lime.



FIGURE 2: SEM micrograph of Portland Cement sample (a) and Pyrex Aggregate (b).

### 3.2 Synthesis of ACC

As proposed in this work, three formulations were made to obtain the ACC: with sodium silicate, potassium silicate and with a mixture of 50% sodium silicate and 50% potassium silicate. The only samples suitable for accelerated test expansion were the AAC basis of sodium silicate (Fig. 3). The other two samples could not be demoulded because a possible lack of polymerization in the AAC synthesis. This suggests that the formulation used was not suitable for the synthesis.



FIGURE 3: Results obtained for prismatic specimens of AAC's: (a) Synthesis with Potassium Silicate, (b) Synthesis with Sodium and Potassium Silicate and (c) Synthesis with Sodium Silicate.

### 3.3 Determination of mortar prism expansion

Expansion patterns were determined for each and every one of the mortars listed in Table 3. The expansion curves for the cement and activated metakaolinite mortars with pyrex aggregate (OPC and MK.S) are given in Fig. 4 (a). A comparison of Fig. 3 shows that for Portland cement mortar, show the greatest expansion compared the metakaolinite mortar. After only 7 days of immersion in the 1M NaOH solution at 80°C, the material had expanded more than the limit allowed in the standard (0.1%).

After 30 days (see Fig. 4 (b and c)), could be seen across the entire surface of the specimen the interaction of the matrix with the pyrex reactive aggregate. It is observed a greater interaction with the alkali-aggregate cementitious matrix enabled, whereas with the OPC mortar base, they are on the surface of the material, showing a detachment of the matrix. The Fig. 4(b). shows that metakaolinite system specimens were in perfect condition at 30 days, with no surface cracks and that they did not exceed the provisions of the standard [35].



FIGURE 4: Expansion curves with the time (a) OPC and MK.S mortars. Image of the mortars (b) MK.S on the 30th day, (c) OPC on the 30th day.

#### 3.4 FRX, XRD and SEM analysis of the mortars

The different mortars were studied with XRD and SEM for mineralogical composition and microstructure after 30 days of storage in the 1 M NaOH solution at 80°C. FRX studies were to determine the composition of the oxides, (see Table 4). Figure 5(a) shows the diffractograms for OPC mortar at 30 days and Figure 5(b) shows the diffractograms for Metakaolinite mortar at 30 days synthesized with sodium silicate, potassium silicate and combination of sodium e potassium silicate. According to these results, it was occurred significant change in the diffractograms, with a substantial decline in the intensity of the phases and appearance of new crystalline phases, for OPC and MK mortars.

The XRD results for the OPC mortars consisted primarily of quartz, alite, belite, portlandite, calcite, chlorite and a few of carbonate (trona and natron). The XRD results of metakaolin mortars showed that they are basically composed of the same crystalline phases of the precursor material with the appearance of phase carbonates, possibly arising from exposure to alkaline solution and interaction with the atmosphere. There was an increase of crystalline phases in materials synthesized with potassium silicate.

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	CaO	TiO <sub>2</sub>	MgO	SO <sub>3</sub>	Others
MK.S	66,09	6,84	0,26	0,31	16,11	7,54	0,06	2,54	0,19	0,06
OPC	52,30	1,79	0,29	1,76	8,04	31,09	0,15	2,69	1,53	0,36



TABLE 4: Chemical composition of the mortars



FIGURE 5: (a) XRD patterns at 30 days of OPC mortars , (b) XRD patterns at 30 days of Metakaolinite mortars (MK.SN – Metakaolinite with sodium silicate, MK.SK – Metakaolinite with potassium silicate and MK.SNK – Metakaolinite with sodium and potassium silicates); Qz quartz; M mica; C3S alite; C2S belite; P portlandite; C calcite; Cl chlorite; Tr trona; Na natron.

Figure 6 shows the SEM micrograph of the samples of mortars OPC based after 30 days of storage in the 1 M NaOH solution at 80°C. It can be seen pyrex aggregate detachment (a) cracking of the matrix (b) and the formation of the expansive gel AAR (c). The Figure 7 shows the SEM micrograph of the samples of mortars metakaolinite based synthesized with sodium silicate after at 30 days of storage in the 1 M NaOH solution at 80°C, where it can be seen that the alkali-activated cementitious matrix is quite porous and presents quartz crystal phases which were not activated, which can contribute to a reduction in the compressive strength of the mortar. Can also observe a strong adhesion between the matrix and the aggregate, different from that observed in the mortar the OPC base.

The large amount of silica in the conditions in which the bars were subjected is a perfect condition for the formation of alkali-aggregate reaction in the structure. However, it can be seen that the bars synthesized AAC did not show the formation of AAR, because in the materials alkali activated the silica and other oxides are very well fixed in the matrix material.



FIGURE 6: SEM micrograph a) spaces coming from the pyrex pearls that took off from the cement matrix, b) cracking of the matrix, and c) formation of the expansive gel AAR of rod-type.



FIGURE 7: SEM micrograph of the samples of mortars metakaolinite a) porosity of the matrix, and b) anchoring of pyrex beads (reactive aggregate).

## 3.5 Results of Ultrasound Analyzes

The mortar bars produced after subjected to the accelerated test of expansion were prepared for analysis by ultrasound. This analysis aims to find potential failures within the material. Figure 8 shows the results of ultrasound tests for mortars based OPC (a) and AAC (b). For mortars ultrasound analysis AAC base (Fig. 8 (a)) may be noted that they had a lower amplitude in relation mortar OPC base, (Fig. 8 (b)) indicating that the CAA has little damaged internal structure. The presence of Alkali-Aggregate Reaction in the mortar bars OPC base, resulted in the expansion of the material causing cracking in bars which was observed in ultrasound analysis at increasing amplitude.



FIGURE 8: a) Analysis of ultrasound mortars AAC base, and b) mortars OPC base.

### 4 DISCUSSION

The deleterious reaction of the concrete induced AAR is a serious problem and that despite the application of several methods that seek to prevent their formation, such as the use of cementitious materials with a low alkaline content, the addition of pozzolanic materials and use of non-reactive aggregate, this problem is increasingly present in structure of concrete [36]. Thus it is appropriate to research presented.

The first aspect to be considered in this study is the high silica content in the precursor material the ACC and the high silica and alkali content in the reactive aggregate. In addition, when evaluated mortar synthesized OPC and AAC based, it is evident that mortars ACC base have much high silica and alkali contents when compared to OPC mortar. Another important aspect is the aggressive conditions of ABNT NBR 15577-4 [35] standard the samples which were subjected, and that even after this submission, AAC samples showed no significant expansion while the OPC samples after 7 days exceeded the value permitted by the standard.

In addition, the interpretation of the results described in this study entailed a review of the role of binder (OPC or AAC) in the formation of expansive AAR products. In the present case, all the conditions required for the formation of expansive products were found (pyrex - reactive aggregate and high silica and alkalis content). The modification of the quartz and portlandite signals in the materials made with Portland cement is a clear indication that the aggregate was attacked by the alkaline solution in which the mortars were immersed. The calcium of cement was incorporated into AAR generated expansive gels.

The SEM study corroborated the formation of alkali–aggregate reaction products for OPC mortar of rod-type (Fig. 6(c)), typical of gels with high sodium content, similar to the findings described by other authors [37]. The AAC systems, on the contrary, tended to form carbonates (Fig. 7 (a)) with high alkali content. However, showed no the formation of the AAR at least 30 days subjected to the aggressive environment in the recommended ABNT NBR 15577-4 standard [35]. The ultrasound studies showed that even subjected to aggressive environments, as proposed by standard, the AAC based mortar preserved their internal structure while the OPC based mortars were damaged, probably due to presence of the AAR.

Although only materials alkali-activated with sodium silicate have been able to be evaluated in this study, new AAC formulations with potassium silicate should be performed, as well as a longer exposure of ACC base mortars to aggressive environment should be carried out to assess more systematically the potential of AAC metakaolinite base as mitigating material from AAR.

### 5 CONCLUSIONS

After all the analyzes and tests performed it can be concluded that the AAC systems based on activated metakaolinite with sodium silicate are less susceptible to generate expansion by AAR than traditional Portland cement systems, even when used reactive aggregates in their composition. This may be related to the fact that ACC although containing high silica and alkalis contend (essential for RAA), the same are entrapped in its matrix and the alkalis available are more likely to form carbonate, while in the cement these components are available to react with water and thus occasioning the AAR. In addition, was ultrasound results corroborated the integrity of internal structure of AAC mortar bar, unlike OPC base mortar.

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