POTENTIAL OF METAKAOLIN IN MITIGATING EXPANSIONS OF ASR IN ACCELERATED TESTS

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Abstract

This paper presents the results of an experimental research concerning the potential of the admixture metakaolin in mitigating expansions from alkali-silica reaction.

Brazilian reactive aggregates were selected for this purpose and accelerated mortar bar tests (AMBT) were performed in order to evaluate ASR expansions over time, and up to 30 test-days. Tests were conducted with a variation on metakaolin contents in order to achieve the optimum content for each situation. Mortar bars were immersed in sodium hydroxide 1N solution at temperature of 80°C. After expansion tests, some microstructural analyses by scanning electron microscope were also undergone to investigate internal ASR products formed and also to search for differences between tested samples.

This paper indicates that metakaolin admixture is able to mitigate ASR expansions, depending on the content tested.

Keywords: Alkali-silica reaction, metakaolin, mitigation, expansion, microstructure.

1 INTRODUCTION

It is well know that all concrete structures will not last forever and long before their service life ends, probably several interventions will have to be made in order to assure enough safety and functionality, so that a huge amount of money and natural resources can be spent to keep these structures useful. However, it is also widely known that in most cases these interventions could have been avoided, minimized or at least postponed if right measures were applied during the manufacture of the concrete, especially when Alkali-Silica Reaction (ASR) is concerned, as this is probably one of the most relevant pathologies found in concrete worldwide.

Many researches show that ASR can be easily mitigated by the use of relatively simple measures such as the right choice of binders and aggregates. Nonetheless, in some cases these measures are difficult or impossible to be put into practice or, sometimes economically infeasible (i.e. change the aggregate), that's where Supplementary Cementitious Materials (SCM) take place, playing an important role in many projects worldwide since the mid-twentieth century, also known as pozzolanic admixtures. First, GGBFS (ground granulated blast-furnace slag) and fly-ash, both residues from metallurgical (or siderurgical) and thermal power plants, were the most important SCM applied in the production of cement or concrete, in order to modify several properties in positive ways to guarantee better performance concerning durability and strength. Their role in the cement paste is quite broad and complex and will not be covered in this paper, except for their main purpose which is to assure longer service life for concrete structure, notably in the prevention of ASR. Several researches have already proved their capability to mitigate ASR expansions, depending on the level of aggregate reactivity as well as the type and content of admixture [1-3].

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In the early eighties, one special type of SCM was developed in order to be an optional alternative to residues from industries and can also offer a controlled product that could meet the growing demand of the civil construction industry. This new controlled SCM was manufacture from natural clays, which were basically transformed into pozzolans by industrial process including calcination and grinding. After a while, it was discovered that one of the best and most effective clays to make this SCM was kaolin, and so Metakaolin was born and started to be used in a widely variety of constructions, not only in concrete, but also in mortar and cement paste. Some previous researches indicate metakaolin as potential admixture in reducing expansions [4-5].

In Brazil, the metakaolin is commonly applied at a maximum content of 14%, as it is the highest amount ever used in a real project, although others would recommend a higher percentage. Several large scale projects have used metakaolin for mitigating ASR in Brazil and in other countries, supplied by the same source, at an *average* of 8% cement replacement such as large dams, infra-structure projects, residential/urban constructions and precast industries. Some of those projects are listed: Irapé Dam (5% of metakaolin - 450.000 m³ of concrete), Capim Branco I & II Dams (6% of metakaolin - 580,000 m³ of concrete), Teles Pires Dam (7% of metakaolin - 1 million m³ of concrete), Pinalito Dam (7.5% of metakaolin - 250,000 m³ of concrete), Cambambe Dam (8% of metakaolin - 50,000 m³ of concrete), and Lauca Dam (10% of metakaolin - 1.5 million m³) of concrete.

Based on the above information, this paper studies the influence of Metakaolin found in the Brazilian Market in mitigating ASR, by using different percentages over cement weight, and also with two different types of reactive aggregates commercially available in Brazil.

2 EXPERIMENTAL PROGRAM

2.1 Materials and Characterization

Due to the vast repercussion of AAR cases in the foundations of buildings in the Northeastern Brazil [6], the selected aggregates were collected from this region. The present study selected two **aggregates** known to the reactive in the field, one from **Metropolitan Region of Recife (MRR)** and the other one collected in the **Metropolitan Region of Salvador (MRS)**.

The **cataclastic granite** studied from **MRR** derives from granitic rocks that underwent a process of metamorphism, and comprises mainly feldspars and quartz, but also containing some mica (sericite and biotite) and having a varied grain size. Its foliation is not well-defined, and the rock is marked by layers of mica and quartz / feldspars porfiroclasts (microcline and plagioclase) stretched out in a mineral orientation. Aggregation of small quartz crystals is noticed as well as its high mineral deformation of all quartz grains. These crystals present undulatory extinction up to 25°.

The aggregate from MRS represents granite in the majority, but it also contains some minor portions with diorite and diabase, all of them representing igneous rocks. But in the same aggregate sampling from the MRS it was also verified from petrographic analyses the presence of typical characteristics from granitegneiss and mylonite (igneous rocks), both in minor portions. Mylonite can be a result from fault shear zones or due to previous rock under high pressures at the borders of granitic mass. Granite-gneiss can also be present just at periphery of the granite mass due to confining stress. Possible explanations for the presence of diabase are: presence of dike that cut off granitic mass or contamination in rock crushing plant. Granite presents an alotriomorphic-granular texture and contains basically feldspars (plagioclase and microcline) and quartz. It is a rock with massive structure, containing plagioclase in the form of irregular crystals, totally saussuritized and also portions with myrmekitic intergrowth. Quartz crystals are also irregular and present strained grains with undulatory extinction up to 30°; amphibole is a restricted mineral. Diorite is similar to granite, except it also contains clinopyroxene and less quartz. Diabase presents an intergranular texture with stretched plagioclase grains and also contains devitrified glass and no quartz grains. The presence of granitegneiss indicates a variation in a portion of the rock related to its texture (granoblastic), structure (oriented) and chlorite-mica aggregates that are preferentially oriented associated with opaques. Mylonite structure is foliated and marked by thin layers of mica and quartz / feldspars stretched out in a slight mineral orientation; quartz is highly deformed (35°).

The **portland cement** used in the study is a Brazilian Cement designed CP II-F-32, with some similarity to ASTM Cement Type I, and its main characteristics are shown in Table 1.

Metakaolin was the selected mineral admixture for this study, being a commercial material available in Brazil and denominated "Metacaulim HP Ultra". Metakaolin is a silico-aluminate pozzolanic admixture, a

product derived from calcination, grinding and micronization of high quality kaolin, according to Brazilian Standard NBR 15894, being a SCM - Supplementary Cementitious Material - with superb pozzolanic activity with most types of cement, especially Portland cement. Its main characteristics are presented in Table 2. Images in Figures 1 and 2 present a microstructural characterization of metakaolin by Scanning Electron Microscope (SEM) with Energy Dispersive Spectroscopy (EDS), X-Ray Diffraction (XRD) and by a Transmission Electron Microscope (TEM). Analyses by SEM were performed on a LEICA equipment (model S440i) using a secondary electron detector (SE) and also EDS for the study of chemical elements. Samples were sputter-coated with gold. Analyses by X-ray diffraction (XRD) were performed on a Siemens equipment (model D5000) with preparation of oriented specimens. For TEM, it was used a JEOL JEM 2100 transmission electronic microscope (TEM) equipped with an Energy Dispersive Spectrometer (EDS) and selected-area electron diffraction (SAED) patterns.

SEM images (Figures 1a,b) indicate irregular morphology particles and the presence of Si and Al, mainly (Figure 1c); X-ray diffraction indicates metakaolin is mainly composed by amorphous material, with some illite (Figure 1d). TEM micrographs presented in Figure 2a show metakaolin nanoparticles in majority as hexagonal shape. The EDS analysis indicated as well only the aluminosilicate. Structures with irregular morphology and crystalline phases were also identified by TEM and can be seen in Figure 2b (in red). The EDS analysis indicated the presence of some magnesium beside the aluminosilicate. For the diffraction pattern (SAED), Figure 2c was obtained from exactly the same area as Figure 2b. The rings presented in Figure 2c indicate an important amorphicity of material at the sample analyzed as well as a crystalline portion due to spots detected. Considering sample was dispersed in 2 ml of isopropanol and sonicated for 10 min and droplets of this dispersion were placed over a copper grid coated with carbon films and dried under air, it is possible this method (TEM) analyzed only metakaolin nanoparticles (< 1 micrometer).

A HRWR **superplasticizer** (high range water reducer admixture) based on polycarboxylate new technology polymer was used in conjunction with the metakaolin substitutions in order to maintain the specified consistency prescribed by standard. Its characterizations show a pH equal to 7.03, solid content of 34.91 % and specific gravity equal to 1.085 g/cm³

2.2 Methods of Analyses

The experimental program involves a study with the admixture metakaolin in partial substitution to cement in the presence of two reactive aggregates in order to test its potential to mitigate expansions from alkali-silica reaction. The AMBT (accelerated mortar bar test) Method, according to ASTM C 1567 (similar to Brazilian Standard, ABNT - NBR 15577, Part 5) [7,8] was performed in order to evaluate ASR expansions over time; the length change of the bars was measured over time up to 30 days. Tests were conducted with a variation on metakaolin contents (from 6%, 10% up to 14%, when necessary), besides the reference sample (with no admixture), in order to identify the best content of this admixture in mitigating ASR. The range of metakaolin percentage studied was based on the real Brazilian applications, according to the comments in the introduction (item 1). Mortars were cast with a proportion of cement to aggregate of 1:2.25 and w/c 0.47. The specimens of each mixture were maintained at temperature of 80 ± 2 °C, and immersed in a sodium hydroxide 1N solution for a period of 30 days.

After expansion tests, some microstructural analyses by the scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS) indicated in 2.1 were also undergone to search for differences between tested mortar samples and investigate internal ASR products formed. Morphology analysis was performed in broken samples using secondary electron (SE1) microscopy and a complementary investigation was done in polished samples using backscattered electrons (QBSD) to observe integrity of aggregates and also transition zone. Microstructural analyses were carried out in mortar samples of reference and those with major ASR mitigating contents of metakaolin, for both aggregates (from MRS and MRR). For backscattered electrons analyses, samples were embedded with epoxy into plastic cylindrical molds followed by vacuum impregnation. The automatic polishing involved several silicon carbide sandpapers and also specific polishing cloths. A nonaqueous lubricant was used during all polish procedure and associating to the cloths, a diamond paste was also used.

3 RESULTS AND DISCUSSION

3.1 AMBT Tests

Figure 3 shows the expansive behavior of mortar bars made with cataclastic granite (from MRR) and three amounts of metakaolin (6, 10 and 14%) besides reference condition. According to the limits (ASTM C33 and NBR 15577/1) [9,10] established for ASTM C-1567 and NBR 15577/5) [7,8], at 16 days the metakaolin replacement level of 14 percent showed average expansion lower than 0.10%. The reference mortar bar and the level of 6 and 10 percent had presented excessive expansions. Along time and up to 30 days all tested conditions had continued to increase expansions; at 14 percent of metakaolin, expansions are still under 0.20%.

The expansion of the mortar bars made with rock from MRS and different levels of metakaolin (0, 6 and 10%) are presented in Figure 4. As expected, the reference sample showed the greatest expansion. At 16 days, 6 percent of metakaolin was able to mitigate expansions, as well as 10 percent, according to standard limit. At 30 days, it is interesting to point out that both tested metakaolin levels (6 and 10%) had achieved the same range of higher expansion (0.23%).

In general, increasing amounts of the admixture of metakaolin resulted in a reduction in the expansion in majority, and no pessimum proportion was observed in the tested conditions of this study. ASR mitigate measures involving metakaolin had been previously indicated in some publications, such as [11,12]. The results from [13] had shown that metakaolin requires at least 10 percent to suppress ASR.

Table 3 summarizes the 16 and 30-days expansion results from AMBT.

According to [5], the high alumina content of metakaolin can improve mitigation of ASR expansion. In the research presented by [5], low levels of metakaolin (10%) were needed to control expansion below 0.1% when compared to the amount of fly ash required to achieve the same result in the presence of high reactivity volcanic sand. The ASR expansions in the study by [4], with a CSA cement in the presence of a moderately reactive siliceous limestone (Spratt Aggregate) presenting 0.44% of expansions at 16 days, could be mitigated by using 10% of metakaolin. On the other hand, with a highly reactive aggregate (and about 0.70%-expansion at 16 days) this admixture content was not proper, although next to the limit.

3.2 Microstructural Analyses

The microscopy analyses in fractured samples by SEM using secondary electrons showed the presence of pores containing cracked and massive gel (mainly calcium sodium silicates with traces of other elements by EDS) in all samples, but the incidence of those gels was clearly seen with more intensity in the reference samples (without admixture) compared to samples with metakaolin admixture, as expected. Some pores contain also crystallized products having rosette morphology. This behavior had been observed by [14] in previous studies with rice husk-ash. Differences in cement pastes were detected during analyses. Metakaolin at highest contents had provided a denser cement-paste matrix in relation to the references samples, without the mineral admixture. Backscattered electrons image analysis indicates that the aggregates cracked substantially after the accelerated test; however, the degree of damage of the aggregate decreased markedly when the level of metakaolin replacement was increased. Gel formations at the transition zone (TZ) are always most intense at reference samples; this characteristic is seen at samples containing admixture, but with more difficulty and only locally at high magnifications. Figures 5 to 12 and 13 to 21 present the main characteristics observed by SEM with secondary and backscattered electrons, respectively.

Some explanation about metakaolin potential to control aggregate deterioration from ASR can be seen in Warner et al. [5] and Chappex and Scrivener [15], considering the aluminum content of this SCM and also the reduction of alkalinity in the pore solution in [15].

4 CONCLUSIONS

The results of the AMBT indicate that both aggregates are reactive (> 0.20% at 16 days in MABA); therefore, measures should be taken to prevent the potential expansion of mortar and concrete made with these aggregates due to ASR. Those results obtained through expansion tests correspond to the reactive mineralogy of aggregates found through petrography.

Incorporating metakaolin reduced the mortar-bar expansion for both aggregates. Replacement levels of 10 and 14 percent of this admixture produced the least expansion (and <0.10%) for granitic rock (from MRS) and cataclastic granite (from MRR), respectively. No pessimum level was found for this admixture, considering the tested conditions for this study.

ASR products appeared in all samples, but the presence of the tested mineral admixture (metakaolin) reduced significantly incidence of ASR gel and also cracking that commonly develops in aggregates containing reactive minerals. Cement-paste matrix of mortars containing metakaolin at ASR optimum contents are denser suggesting a minor ingress of alkalis from solution and thus less expansion.

Metakaolin tested in the present study had presented a good performance at certain contents and had shown to be an interesting SCM in mitigating ASR from some Brazilian reactive aggregates.

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TABLE 1: Chemical (%) and physical characteristics of the cement CP II-F-32.

Silicon dioxide (SiO2)	20.07	Loss of ignition	3.82	Specific gravity (g/cm ³)	3.15
Aluminum oxide (Al2O3)	4.26	Insoluble residue	1.68	Blaine fineness (cm ² /g)	3995
Iron oxide (Fe2O3)	3.71	Sodium oxide (Na ₂ O) – totals	0.14	Residue on sieve # 200 - 75 μm (%)	0.94
Calcium oxide (CaO)	61.56	Potassium oxide (K ₂ O) – totals	0.79	Residue on sieve # 325 - 45 μm (%)	7.76
Magnesium oxide (MgO)	2.91	Alkaline equivalent (Na2Oeq)* - totals	0.66	Average size of the grains	0.05
Sulphur trioxide (SO3)	2.86	Autoclave expansion (%)	0.04	(µm)	9.93

* $Na_2O_{eq} = \%Na_2O + 0.658 . \%K_2O$

TABLE 2: Chemical (%) and physical characteristics of metakaolin.

Silicon dioxide (SiO ₂)	51.43	Loss of ignition	1.28	Specific gravity (g/cm ³)	2.58
Aluminum oxide (Al ₂ O ₃)	42.53	Potassium oxide (K2O) – totals	0.95	BET fineness (m ² /g)	26.75
Iron oxide (Fe ₂ O ₃)	2.00	Sodium oxide (Na ₂ O) – totals	0.02	Average size of the grains (µm)	9.20
Titanium oxide (TiO ₂)	1.07	Zirconium oxide (ZrO ₂)	0.03	Chanelle Dounclanisity	
Calcium oxide (CaO)	0.16	Phosphorus oxide (P2O5)	0.04	Index (mg of Ca(OH) ₂ / g	1,440
Magnesium oxide	0.28	Moisture	0.59	of sample)	

Note: Chapelle Pozzolanicity Index was determined by ABNT NBR 15895, following Raverdy, M.; Brivot, F.; Paillere, A.M.; Dron, R. - Appréciation de l'activité pouzzolanique des constituants secondaires. 7e. Congrès International de la Chimie des Ciments, Paris, 1980. Limit to consider a pozzolanic material: > 750 mg of Ca(OH)₂/ g of sample, according to NBR 15894.

TABLE 3: Expansions	at 16 and 30	days (%) in	the AMBT.
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Replacement	0%	6%	10%	14%	
Aggregate 1 - MRR					
16 days	0.21	0.13	0.11	0.08	
30 days	0.40	0.26	0.24	0.17	
		Aggregate 2 - MR	S		
16 days	0.21	0.09	0.07	-	
30 days	0.30	0.23	0.23	-	





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EHT = 20.00 KV Detector = SEI Mag = 7.50 KX by
Detail of metakaolin and very fine phase grouped.





d) Amorphous bands and some peaks relative to illite.

c) Typical EDS Spectrum of metakaolin.

FIGURE 1: c) SEM images of metakaolin; d) XRD of metakaolin.



FIGURE 2: a), b) Transmission electron microscopy (TEM) images of metakaolin; c) selected area electron diffraction (SAED) pattern.



FIGURE 3: Expansions from AMBT tests - Aggregate 1, from MRR.



FIGURE 4: Expansions from AMBT tests – Aggregate 2, from MRS.



Eletrobras EHT=2000 KV Detector=SE1 Mag= 5.00 KX FUTTORS FIGURE 5: Cement paste of mortar with MRS aggregate – without metakaolin.



Furnas FIGURE 6: Cement paste of mortar with MRS aggregate – with metakaolin at 10%.



Eletrobras Furnas FIGURE 7: Cement paste of mortar with MRR aggregate – without metakaolin.



Electrobras EHT = 20.00 KV Detector = SE1 Mag = 5.00 KX 20µm Furnas FIGURE 8: Cement paste of mortar with MRR aggregate – with metakaolin at 14%.



FIGURE 9: Cracked ASR gel in the mortar with MRS aggregate – without metakaolin.



FIGURE 11: Pores containing cracked massive gels in the mortar with MRR aggregate without metakaolin.



FUTNAS EHT = 20.00 KV Detector = SE1 Mag = 350 X FUTNAS EHT = 20.00 KV Detector = SE1 Mag = 350 X FUTNAS ENTRY AND A SECOND HARD AND A SEC



SElectrobras EHT = 20.00 KV Detector = SE1 Mag = 250 X FURDAS FURDAS ET Cracked massive ASR gel in the mortar with MRR aggregate – with metakaolin at 14%.



Selectrobras Furnas FIGURE 13: Cracked aggregate particle in the mortar with MRS aggregate - without metakaolin.



FIGURE 15: Cracked (c) aggregate particle in the mortar with MRR aggregate - without metakaolin.



FIGURE 14: Intact aggregate particles from mortar containing MRS aggregate and metakaolin at 10%.



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FIGURE 17: Cracked aggregate (A) and cement matrix with gel (g) and great microcracking next to a rosette (R) ASR products filling a pore - mortar with MRS aggregate, without metakaolin.



FIGURE 18: Transition zone with surface of aggregate completely deteriorated next to a massive gel (g) in pore - mortar with MRS aggregate, without metakaolin.



Selectrobras EHT = 2000 KV Detector = OBSDMag = 1.70 KX FUTTAS FUTTAS TZ with some crack on aggregate next to a pore (P) with ASR product - mortar with MRS aggregate and metakaolin at 10%.



Eletrobras EHT = 2000 KV Detector = OBSDMag = 900X FORMATION ENTRY AND A COMMENT OF THE PROVIDENCE OF



Eletrobras Furnas FIGURE 21: TZ with some crack on aggregate and cement paste - mortar with MRR aggregate and metakaolin at 14%.