# ELEMENTAL AND PHASE CHARACTERIZATION OF COARSE AGGREGATES COMMERCIALLY AVAILABLE FOR CONCRETE PRODUCTION IN THE BRAZILIAN NORTHEAST

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#### Abstract

Coarse aggregates are the main component by mass of concrete mixes. They are often considered inert in many applications as far as concrete durability is concerned. When alkali aggregate reaction is expected, special attention must be given to the aggregates, cement blends and environment interactions. Most standards limit the total amount of alkali per cubic meter of concrete to only few kilograms. The Portland cement itself should contribute with not more than 1% of its mass. This is because of the widely known degrading and expansive mechanism involving the precipitation of AAR gels. Hence, it is important to account for certain chemical components and their crystal structure when assessing potential susceptibility to expansive mechanisms, as many minerals are acknowledge to have greater susceptibility to AAR. This paper aims to evaluate the elemental composition and the crystalline phases in several commercially available coarse aggregates from the Northeast area of Brazil. Samples from around fifty commercial producers were selected. The samples were ground and sieved through a 45µm sieve. The chemical composition was evaluated by means of x-ray florescence (WDS) and the crystalline phases by means of x-ray diffraction (Bruker D2-Lynx Eye detector). Considerations are given about the total alkaline silicate composition as well as for the type of each crystalline phases with respect to their silicate structure.

Keywords: ARR; ASR; CHESF and Micro analytical techniques.

#### 1 INTRODUCTION

This paper presents preliminary results as part of a 4-year's comprehensive research, development and innovation proposal (R&D+I) with CHESF-Comapnhia Hidroletrica do São Francisco [1]. CHESF is the biggest electric company in the Northeast of Brazil (10.6Gw), mostly from hydroelectric but also from thermal and wind sources. Half of its 14 hydroelectric dams present long-term developed problems (extensive cracking and damaged concretes promoted by steel corrosion and other complex chemical mechanisms in different levels) [2].

As far as alkali aggregate reaction is concerned, standards highlight the desirable characteristics of aggregates, which is mostly based on their chemical (equivalent alkali content) as well as mineral composition [3]. However, the aim to improve concrete durability in a long-term is not an easy task, especially for the constructions of large dams [4,5]. One of the main problems is to select aggregate sources from a logistically and financially effective source, which is obviously dependent on geological features.

Most standards recommend the use of ground coarse aggregates in alkaline solutions in methodologies to assess susceptibility to AAR [3]. Hence, this paper concerns with the variability of how alkalis are distributed within each rock microstructure. Preliminary results aim to improve the evaluation of the actual susceptibility to AAR of the aggregates by comparing coarse aggregate alkali distribution and its effect on finely ground methodology assessments.

### 2 MATERIALS AND METHODS

Coarse aggregate samples were collected from more than 50 sources around six states of the Northeast of Brazil. The samples were ground and sieved through a 45 $\mu$ m sieve prior to testing. The chemical composition was evaluated by means of x-ray florescence (WDS-XRF 1800/Shimadzu) and the crystalline phases by means of x-ray diffraction (CuK $\alpha$ -Bruker D2-Lynx Eye detector). The microstructure of rocks were performed in polished sections (down to 1/4 $\mu$ m) in a Quanta 450 scanning electron microscope with energy dispersive X-ray (EDAX). Spot analyses and dot mapping were performed in 20kV, using ZAF correction [6].

# 3 RESULTS AND DISCUSSION3.1 Chemical composition

Figure 1 shows that the majority the aggregates chemical composition had similar  $Na_2O$  equivalent and  $SiO_2$  contents of c.a 7.0 % and 70.0% in average, respectively. It was found that the increase of alkali content occurred with a reduction of the silica content almost in a linear fashion (Figure 2). Figure 3 shows the classification with respect to the main two multicationic parameters of each aggregate [7]. It can be observed that most samples fell within the composition between granodiorite and diorite monzodiorite.



FIGURE 1: XRF chemical composition of the coarse aggregates.



FIGURE 2: XRF chemical composition of coarse aggregates in milications.



FIGURE 3: Multicationic characterization R1R2 of coarse aggregates [7].

# 3.2 X-ray diffraction

Figure 4 shows the XRD pattern of three samples selected among the categories from granite to monzodiorite/gabrodiorite. Table 1 lists the main crystalline phases in coarse aggregates of the Northeast area of Brazil. It can be seen that alkalis are present in at least four main phases (microline, albite, muscovite, phoglopite and cordierite).



FIGURE 4: XRD pattern of three coarse aggregate samples. (M): muscovite;(C): cordierite; (Mi): microcline; (Q): quartz; (A): albite.

Sample code	Quartz	Carbonates	Feldspar	Micas	Other
A1		-	√(Sodalite)	-	-
B1			-	-	-
C1		-	√(Albite)	-	-
D1	$\checkmark$	-	√(Kalsilite)	√(Phlogopite)	√(Reyerite)
E1					
F1	$\checkmark$	√ (calcite magnesian)			
G1	$\checkmark$	√ (calcite magnesian, Dolomite)			
H1	$\checkmark$	√ (calcite magnesiane, calcite)			
II	$\checkmark$	√ (calcite magnesiane, Dolomite)			
A2		-	√(Albite)	√(Phlogopite)	√(Nefertisite)
B2	$\checkmark$	-	√(Albite)	√(Phlogopite)	√(Laumonitite)
C2	$\checkmark$	$\sqrt{(\text{Dolomite})}$	√(Albite,Sodalite)	√(Musvoite)	√(Sodalite)
D2	V	√ (calcite magnesian)	√(Albite)	√( Phlogopite)	√(Cordierite)
E2	$\sqrt{+}$ Cristobalite	-	√(Anordite,Sodalite)	√(Muscovite)	-
F2	$\checkmark$	-	√(Albite,Leucite)	√(Muscovite)	√(Aenigmatite)
G2	$\checkmark$	√ (calcite magnesiane,Do lomite)	√(Albite)	√( Muscovite)	√( Aenigmatite)
H2	√+ Cristobalite	$\sqrt{(\text{Dolomite})}$	√(Albite)	√( Biotite)	√( Aenigmatite)
I2	$\checkmark$	-	√(Albite,Sodalite)	√( Phlogopite)	√(Ferroglaucophane)
J2	√+ Cristobalite	$\sqrt{(Dolomite)}$	√(Albite)	√( Phlogopite)	√( Ferroglaucophane)
K2	$\checkmark$	-	√(albite,Nefeline)	√(Muscovite)	
A3	$\checkmark$	-	√(Albite,Microclin e)	√(Muscovite, Phlogobite)	-
B3	$\checkmark$	-	√(Microcline)	√(Muscovite)	√(Aenigmatite)
C3	$\checkmark$	-	√(Albite,Microcline)	√(Muscovite)	-
D3	$\checkmark$	-	√(Albite)	√(Muscovite)	√(Clinochlora)
E3	$\checkmark$	-	√(Albite,Microcline)	√(Muscovite)	√(Pargasite)
F3		-	√(Albite,Microcline)	√(Muscovite)	-
G3		-	√(Albite,Microcline)	√(Muscovite)	-
H3		-	√(albite,Nefeline)	-	-
I3	$\checkmark$	-	√(Albite,Microcline)	√(Muscovite)	-

TABLE 1: Crystal phase assemblages in coarse aggregate of the Northeast area of Brazil.

## 3.3 Scanning Electron Microscopy and Dot Mapping

Figure 5 shows *SEM-BEI* images of the four selected aggregates samples as presented in Figure 3. Only aggregate sample 03 presented lower equivalent alkali whereas the others presented similar values above 6% (see Fig.3). It can be observed that the occurrence of cracking, mostly at the interface of the phases but also through grains. The distribution of alkali occurred in two particular fashions: (i) clusterized in larger grains constituted of K or Ne with Si (sample 03) and (ii) more distributed in smaller grains or veins with alternated predominance of K or Ne (other samples).



FIGURE 5: SEM-BEI images of four selected coarse aggregate samples. (01): Granite; (02): Granodiorite; (03): Granite; (04): Monzodiorite/Gabrodiorite (see Fig. 3).

As far as grey scaling is taking into account, the samples presented three to four phases plus defects (dark features). Samples 01 and 03 presented a more circular granular nature, samples 02 and 04 showed elongated features. Figure 6 shows the segmentation of the BEI images (Fig. 5), using *Quantum Particle Swarm Optimization Multilevel Thresholds*, as proposed by Queiroga et. al [8].



FIGURE 6: Images segmentation of four selected coarse aggregate samples. (01): Granite; (02): Granodiorite; (03): Granite; (04): Monzodiorite/Gabrodiorite (see Fig. 3). (light blue): Si-Ne containing phases; (yellow): Si-K containing phases; (dark blue): Si containing phases plus defects; (red): Fe containing phases [8].

It was observed that the distribution of alkalis is, as observed in these four samples, predominantly within Si-Ne containing phases (albite), followed by Si-K containing phases (microline, phlogopite and muscovite). The amount of defects (between 12.7% and 13.4%) was consistently higher in samples 01 and 02, mostly within grain boundaries, either in clusterized or dendritic occurrences. In samples 03 and 04, the level of defects were much lower (less than 1%), mainly throughout the grains themselves.

## 4 GENERAL REMARKS

This paper presents preliminary results of the elemental composition and the crystalline phases in several commercially available coarse aggregates from the Northeast area of Brazil. The aggregates elemental composition fell within the composition of granodiorite with occurrences of monzonite, diorite ad tonalite groups. The total alkali content varied between 5.0 and 10%, with equivalent Na<sub>2</sub>O of 7.5% in average. The higher the content of SiO<sub>2</sub>, the lower the total alkali content. The main alkaline phases were Si-Al such as albite and Si-K containing phases such as microline, flogobite and muscovite. The use of PSO multivariable threshold technique [8] was able to perform automatic image analyses of the aggregates. Further analyses will cover a larger number of samples of the northeast area of Brazil with consideration on the solubility of each phase in alkaline media as well as their susceptibility to cause expansion in mortars and concrete.

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## 6 **REFERENCES**

- [1] Silva, A.C., Torres, S.M., Vieira, A.P., Cavalcanti, A.J.C.T., Silva, P.N., Andrade, T.W.C.O.(2016): Potential of non-destructive and micro analytical techniques applied to the study of damage assessment and mitigation in asr prone structures. *15th international conference on alkali-aggregate reaction*, São Paulo-Brazil (unpublished).
- [2] EPE Empresa de Pesquisa Energética (2013): Ministério de Minas e Energia do Brasil. *Anuário Estetístico de Energia Elétrica*, Rio de Janeiro.
- [3] Swamy, R.N. (1992): The Alkali-Silica Reaction in Concrete. Glasgow/London: Blackie / New York: Van Nostrand Reinhold.
- [4] Batista, D.G.(2013): Investigação da deterioração de concretos de UHEs por reações com os agregados. Tese de Doutorado, Universidade Federal de Goiás, Goiana, Brasil.
- [5] Hasparyk, N.P.(2005): Investigação de concretos afetados pela reação álcali-agregado e caracterização avançada do gel exsudado. Tese de Doutorado, Universidade Federal do Rio Grande do Sul, Porto Alegre, Brasil.
- [6] Jones, I.P.(1992): Chemical microanalysis using electron beams. Ed. I.P. Jones. London: The Institute of Materials, 241.
- [7] H. de la Roche, J. Leterrier, P. Grandclaude and M. Marchal (1980): A classification of volcanic and plutonic rocks using r1r2-diagram and major-element analyses -- its relationships with current nomenclature. *Chemical Geology*, 29, 183–210.
- [8] Queiroga, E.; Cabral, L.A.F.; Vieira, A.P.; Lima Filho, M.R.F.; Gomes, K.C.; Torres, S.M (2016): Quantum particle swarm optimization applied to automated crack pattern assessment of backscattered electron image in cementitious systems. *15th international conference on alkaliaggregate reaction.* São Paulo-Brazil (unpublished).