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# Investigation of Alkali-Aggregate Reaction in Carbonatic Rocks

# Investigação da Reação Álcali-Agregado em Rochas Carbonáticas

**A. L. Z. P. SILVEIRA** [a]
liviazeitune@hotmail.com

**E. M. GUIMARÃES** [b]

**S. L. R. LENHARO** [c]

**N. P. HASPARYK** [d]

**W. C. CHIOSSI** [e]

**R. M. BITTENCOURT** [f]

## Abstract

Alkali-aggregate reaction (AAR) may occur in concrete involving siliceous or carbonate rocks and alkaline hydroxides. Depending on the type of minerals present in the rock, it is possible to cause alkali-silica/silicate and alkali-carbonate reactions. The preliminary study of these rocks is an obligatory procedure before their application in a concrete structure. This paper presents a laboratory study with the main purpose of verifying the potential in causing alkali-silica/silicate or alkali-carbonate reactivity of carbonate rocks from Goiás and Mato Grosso states and Distrito Federal surroundings. With this approach, several standard methodologies in the AAR investigations and a detailed mineralogical analysis of the mentioned rocks are showed and the tests results discussed.

***Keywords:*** carbonate rocks; reactive potentiality; alkali-silica reaction; alkali-silicate reaction; alkali-carbonate reaction.

## Resumo

A reação álcali-agregado pode ocorrer no concreto envolvendo tanto rochas silicosas como rochas carbonáticas e os hidróxidos alcalinos. Dependendo do mineral presente nas rochas, podem ser desencadeadas reações do tipo álcali-sílica/silicato e/ou álcali-carbonato. O estudo preliminar destas rochas, antes de seu emprego, torna-se um procedimento obrigatório. Este trabalho apresenta um estudo realizado em laboratório com o objetivo de verificar a potencialidade reativa de rochas carbonáticas provenientes do estado de Goiás, Mato Grosso e entorno do Distrito Federal, e possíveis causadoras de reações tanto do tipo álcali-sílica/silicato como do tipo álcali-carbonato. Desta forma são apresentadas diversas metodologias de investigação através de ensaios normalizados e análise criteriosa da mineralogia destas rochas no que diz respeito à sua reatividade.

***Palavras-chave:*** rochas carbonáticas; potencialidade reativa; reação álcali-sílica; reação álcali-silicato; reação álcali-carbonato.

[a] *MSc., Geologist of CNEC Engenharia S.A.*
[b] *PhD., Geologist and Professor of Geoscience Institute, Brasilia University – UnB.*
[c] *PhD., Geologist of Federal Police.*
[d] *PhD., Civil Engineer of Support and Technical Control Department, FURNAS CENTRAIS ELÉTRICAS S.A.*
[e] *Specialist, Chemist, Ipiranga Quimica S.A.*
[f] *Civil Engineer, Manager of Support and Technical Control Department, FURNAS CENTRAIS ELETRICAS S.A.*

## 1 Introduction

The alkali-aggregate reaction (AAR) is a chemical process in which some components of the aggregate react with dissolved alkaline hydroxides in concrete solution. These reactions are divided in three types: Alkali-Silica Reaction (ASR), Alkali-Silicate Reaction and Alkali-Carbonate Reaction (ACR).

The occurrence conditions of AAR are not well-known, once that the interaction of several factors may occur in order to speed up or even to inhibit the reaction [1]. The AAR may occur in matter of days or after several years [2].

Studies on ACR mechanisms were first began in the fifties, mainly by the Chinese, due to deterioration problems on concrete structures that had the carbonate rock as aggregate. These studies were not quite successful and soon the interest for the subject was diminished. However, through the recent deterioration evidences on concrete with carbonate rock as aggregate in dams, the need for new studies was verified.

According to Ozol [3], several reactions involve carbonate rocks, which can be dolomite or non-dolomite, embedded in concrete or mortar. However, only one reaction is recognized, what is the expansion of the dedolomite reaction. This reaction may represent a great problem in concrete of dams.

It is important to emphasize that carbonate rocks can develop silica/silicate reactions (ASR) as well, when they have silicate in their composition. It is believed that clay minerals such as illite, for example, may also contribute to development of the expansion, according to Kihara [4]. According to Paulon [5], carbonate rocks with 10 to 20% of clay can be considered reactive. Thus, the reaction of dedolomite, the disorganization of calcareous texture and the presence of clay minerals, which becomes easier the desegregation of aggregate, can cause a combination of events that leads to the AAR expansion.

This study is focused on the AAR investigation in order to study different carbonate aggregates and to understand how these different rocks behave during tests and analyses to identify their ASR and ACR potential.

Up to today there are considerable divergences on the possible reaction mechanism. Several hypotheses have been enunciated. The alkali-carbonate reaction is a quite complex reaction and the lack of specific and safer lab tests created the need and the interest for this research. This study also considers new studies about alkali-carbonate reaction, not only focusing on the dedolomite process, only to understand better and find out the factors that cause this reaction.

## 2 Experimental program

Carbonate rocks with different composition, texture, structure and provenience were selected and submitted to several AAR analyses and tests. One of the carbonate rocks selected for this study is an aggregate used in concrete of a dam.

### 2.1 Carbonate Rocks – Aggregate

The selection of carbonate rock samples was done to collect materials with different mineralogical and texture characteristics, as wide as possible in order to evaluate their behavior as aggregate and the possible variations of this behavior. Amongst the samples, there are rocks from quarries identical to those considered for a dam built more than thirty years ago (Table 1).

### 2.2 Investigation Methods

The investigation methods considered in this research were selected with the aim to verify the behavior of carbonate rocks used as aggregate, as well as their reactions with the cement paste. Firstly, the rocks were assessed in their natural state and then they were investigated through several methodologies based on standards, described as follows.

#### 2.2.1 Characterization of carbonate rocks in their natural state

The texture, mineralogical and chemical characteriza-

**Table 1 – Provenience, litologic classification and lithostratigraphic unit of the samples**

| SAMPLE | PROVENIENCE (STATE) | LITOLOGIC CLASSIFICATION | LITHOSTRATIGRAPHIC UNIT |
|---|---|---|---|
| 284 | Vila Propício Area (GO) | Very fine Dolarenite | Brasília Zone – Paranoá Group |
| 285 | Vila Propício Area (GO) | Recrystallized Calcareous | |
| 286 | Distrito Federal Surroundings | Calcilutite | |
| 289 | Portelândia (GO) | Calcilutite | Sedimentar Paraná Basin – Irati and Corumbataí Formations |
| 290 | Alto Garça (MT) | Bio-Calcilutite Dolomite | |
| 291 | Alto Garça (MT) | Bio-Calcilutite Dolomite | |

tion of the selected carbonate rocks were evaluated through petrographic, mineralogical, difratometric and chemical analyses.

**a) Petrographic Analysis – Optical Microscopy (OM)**

In addition to the information on the ASTM C-295 [6] standard, the petrographic analysis was accomplished in accordance with the conventional techniques of microscopical analysis for determination of texture and composition characteristics of the rocks.

Microscopical petrographic analyses were performed using the transmitted light optic microscope, with the aim to classify the rock and estimate its mineralogy, mostly in what refers to carbonates and silicates. By this stage, the concern was to perform a representative sampling; therefore, 18 impregnated thin sections were prepared.

**b) X-Ray diffraction**

The identification of the crystalline phases of the carbonate rocks used as aggregate was done through X-Ray diffraction (XRD) technique. Thus, it was considered a complementary evaluation to the petrographic classification of the rocks with this analysis. The samples were submitted to a previous grinding (100% under # 400) to provide representative fine size material. Part of this material was submitted to Powder Diffraction X-Ray Analysis (Integral Sample).

Then, the clay fraction of these samples was separated by sedimentation method, what was followed by the preparation of 3 samples by using oriented glass thin sections. The one which contained the "sample" for orientation was smoothly slided by another thin section. This method is called Oriented X-Ray A nalysis.

One of these clay fraction samples was dried in air before analysis. The second one had ethileneglycol saturation and the third one was submitted to calcination at 550ºC for 3 hours before submission to Oriented X-Ray Analysis

**c) Chemical Analyses**

The chemical analyses were performed according to complexometric method to obtain Al, Ca, and Mg oxides and gravimetrical method to determine siliceous oxide and insoluble residue. The objective of this analysis was to quantify the chemical elements in the aggregates and to correlate them to the potential reactivity of the rocks.

### 2.2.2 Investigation of ASR on carbonate rocks

For the investigation of ASR on the carbonate rocks, the accelerated method in mortar bars according to ASTM C-1260 [7] was used, through 25 mm x 25 mm x 285 mm dimension specimens.

The bars were immersed in 1N sodium hydroxide solution at 80 ºC for 30 days. It was cast three mortar bars with each one of the selected rock types, as fine aggregate, considering the cement:aggregate proportion of 1:2.25 and w/c ratio of 0.47. The grading requirements of artificial sands, crushed from rocks, followed the ASTM C 1260 [7]. The cement contains a high alkaline equivalent ($Na_2Oeq$), representing 0.88% and 0.70% of total and soluble alkalis, respectively.

### 2.2.3 Investigation of ACR on carbonate rocks

For ACR investigation expansion tests were performed according to the Brazilian standard - NBR 10340/88 [8] (similar to ASTM C 586 [9]), the American standard ASTM C 1105/95 [10], and the Canadian standard AAR 23.26-A/04 [11], as follows:

**a) Rock-Cylinder Method- NBR 10340 [8] and ASTM C-586 [9]**

For this test, cylindrical cores were drilled from rock, with 9 mm of diameter and 35 mm of length. They were immersed in distilled water until it had variation inferior to 0.02% of length. Then, they were immersed in a 1N sodium solution and kept at room temperature for approximately 365 days. The purpose of this test is following up the length variation of the cores and, consequently, being possible to determine the expansions presented by the rock.

NBR 10340 [8] standard does not keep an expansion limit, but according to ASTM C-586 [9], 0.10% of expansion indicates chemical reaction; this is a preliminary test and other tests in concrete should be accomplished for a better characterization of the potentiality of the rock with respect to the ACR by the ASTM C-1105 [10].

**b) Concrete Prism Method – ASTM C 1105 [10]**

This test method - ASTM C 1105 [10] - allows the evaluation of the reactivity potential of the carbonate rocks by the measurement of the length variation of concrete prisms with aggregates from those rocks. It is important to know that the fine aggregate should not be potentially reactive with the alkalis, according to the aforementioned standard, in a case of reactivity potential investigation of coarse aggregates. Thus, a non-reactive quartzes sand was chosen for the tests. The concrete prisms were molded based on a reference mixture used in a concrete dam produced with carbonate rock [12].

Figure 1 – CaO/MgO ratio versus Al2O3 – AAR 23.26 – A standard (11)

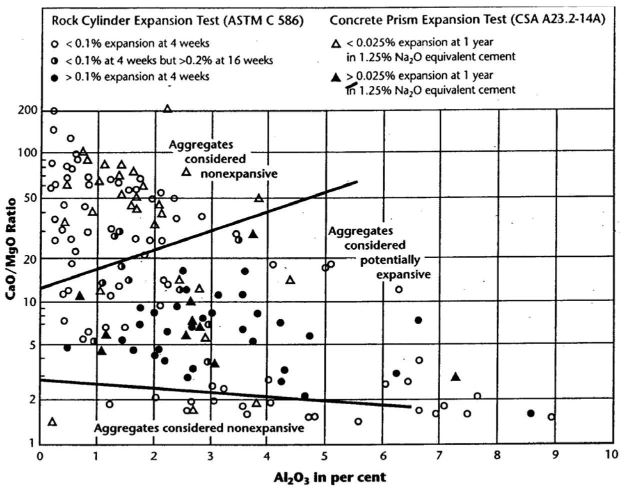


Table 2 – Summary of the petrographic, chemical and XRD analysis of the studied rocks

| SAMPLE | LITOLOGIC CLASSIFICATION | CHEMICAL ANALYSIS | | | | | XRD* | | |
|---|---|---|---|---|---|---|---|---|---|
| | | CaO | MgO | $SiO_2$ | $Al_2O_3$ | RI | M | I | T |
| 284 | Very fine Dolarenite | 32.16 | 16.65 | 3.46 | 1.28 | 5.39 | Do | Ca | Q, Il, Sap |
| 285 | Recrystallized Calcareous | 50.66 | 0.62 | 3.50 | 0.52 | 4.08 | Ca | - | Q, Il, Sap, Do |
| 286 | Calcilutite | 41.12 | 2.71 | 12.84 | 3.63 | 18.86 | Ca | - | Q, Il, Chlo, Do |
| 289 | Calcilutite | 31.34 | 16.04 | 19.13 | 0.38 | 37.46 | Ca | Ant | Q, Do |
| 290 | Bio-Calcilutite Dolomite | 24.30 | 7.67 | 27.35 | 6.25 | 38.20 | Ca | Do, Q | Sap, Il |
| 291 | Bio-Calcilutite Dolomite | 47.44 | 3.28 | 5.48 | 0.84 | 7.15 | Ca | Do | Q, Il, Sap |

*** Legend of XRD analysis:** total sample: (M) major components, (I) lesser components, (T) trace composer and (-) absence of the mineral. Ca: calcite; Do: dolomite; Q: quartz; Sap: saponite; Il: illite; Chlo: chlorite; Ant: antigorite.

The rocks used as aggregate were crushed according to NBR 9941/87, with maximum dimension size of 19 mm (ASTM C 1105/95 [10]). The studied mixture has cement content equal to 295 kg/m³ and w/c ratio of 0.57. The sand rate was, approximately, 34% in mass. It was considered 0.4% of superplasticizer admixture, lignosulfonate-based. Three concrete prisms were prepared, according to ASTM C 157 standard, with dimensions of 75 mm x 75 mm x 285 mm, for each type of selected rock. During the test, the prisms were kept in a moist room, according to ASTM C 511, without immersion in water, being held monthly length readings up to 1 year, and results expressed in % of expansion from the average of the three prisms.

**c) Chemical Method - AAR 23.26-A [11]**

The ACR potentiality of the rock that relates the ratio CaO/MgO versus $Al_2O_3$, proposed by Roger [13] and standardized in the AAR 23.26-A [11], shows the distribution of the studied samples according to its chemical composition and plotted in the graph proposed by the researcher. It was identified two regions for aggregates considered non-reactive and one are defined for aggregates considered potentially expansive, according to Figure 1.

## 3 Results and discussions

The results presented are related to the specific studied materials, which were submitted to instrumental analyses for mineralogical and technological characterization and to reactivity tests.

### 3.1 Rocks Characterization

The results from the petrographic, chemical and X-Ray Diffraction (DRX) analyses are presented in Table 2.

From the analyses performed in the 6 rock samples, 4 different lithotypes are verified: very fine dolarenite, recrystallized calcareous rock, calcilutite and bio-calcilutite dolomite. The calcite presents as major composer, in its majority, followed by the dolomite. The presence, although is small (trace), of silicates is constant in all analyzed rocks, giving emphasis to quartz, saponite, illite, chlorite and antigorite. The chemical analyses corroborate its presence, as indicated in contents of $SiO_2$, $Al_2O_3$ and insoluble residue.

Gillott [14] carried out several petrographic analyses in reactive carbonate rocks and concluded that only those rocks with special texture were reactive in the alkali-carbonate reaction; certain argillaceous dolomite calcareous rocks from Kingston, Canada, were highly reactive. These rocks contain 40 to 60% of dolomite or calcite and about 5 to 10% of insoluble residues, and are composed by crystals of microcrystalline dolomite, about 40 to 50 μm, which are distributed in a microcrystalline matrix of calcite and clay. The rocks evaluated in this work presented a mineralogy composed by calcite, dolomite and clay, as observed by the researcher aforementioned.

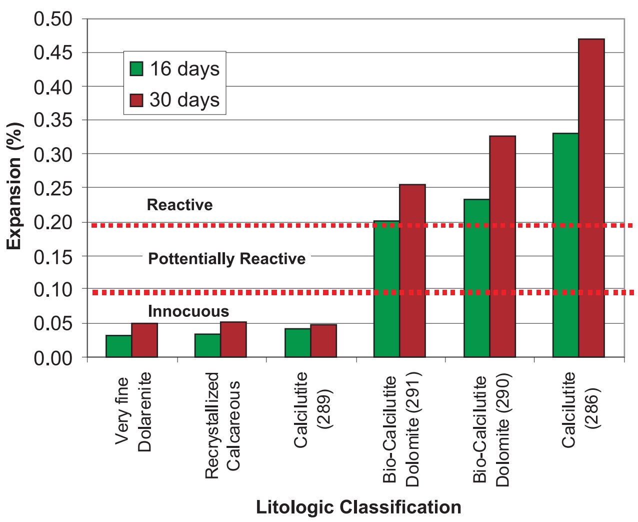
According to Paulon [5], the ACR is intense when the calcite and dolomite are present in equivalent amounts or both are finely divided. Equivalence between them (ratio of 1:1 of calcite:dolomite) was not observed in the samples of the studied rocks.

### 3.2 Expansion Tests

#### 3.2.1 ASR Investigation - Accelerated Mortar Bar Method - ASTM C-1260 [7]

Figure 2 presents the expansion results by the age of 16 and 30 days of the carbonate rock samples.

**Figure 2 – Potential Reactivity Graph – ASTM C 1260/01 Method**




According to ASTM C –1260 [7], expansion up to 0.20% in 16 days means reactive behavior of the aggregate. Yet, for expansions below 0.10%, by the same age, the aggregate is considered innocuous. In case of expansion presents between 0.10% and 0.20%, by the age of 16 days, the expansion will be able to denote behavior either innocuous or reactive. For that, it is necessary complementary information of materials and follows up the expansions until the age of 30 days.
From the test limits presented by the standard, it is observed reactive behavior, by the age of 16 days, for 2 aggregates with dolomite bio-calcilutite lithology and 1 aggregate with calcilutite lithology. It is important to mention that the expansions do not cease at 16 days, they keep up until 30 days after the begging of test. The remaining rocks presented innocuous behavior related to ASR.
Thus, the ASTM C-1260 [7] method showed that even though the rock is carbonate, and is able to originate reactions of the alkali-carbonate type, it can behave as ASR reactive, by this method, once that it presents in its silicates composition, as already observed by their petrographic characterization (item 3.1).
Analyses performed through scanning electronic microscopy (SEM) and EDX, the origin of the expansions (ASR) was corroborated by the samples of the mortar bars with fracture surfaces, once it was possible to verify the presence of AAR neoformed products, with typical morphology and alkaline calcium siliceous composition. The following figures show one of the identified products. Additional Information can be found in Silveira [12].

**Figure 3 – SEM micrograph of mortar bar sample 291B. Void filed with cracked massive gel containing in the center a botrioidal texture**

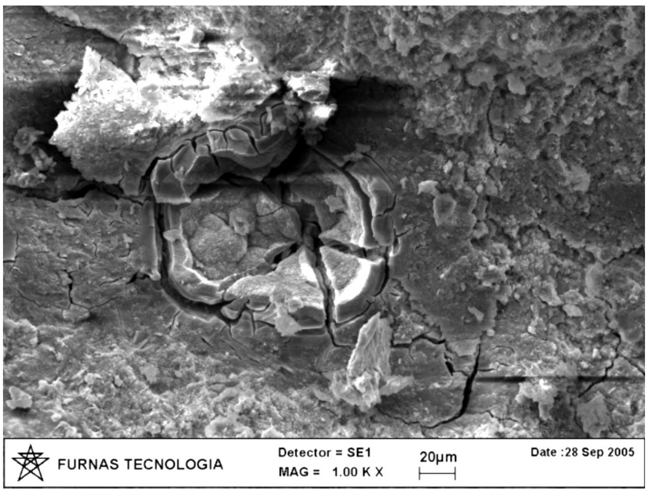
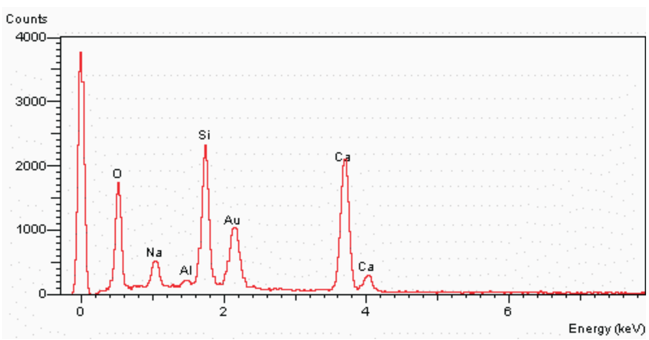


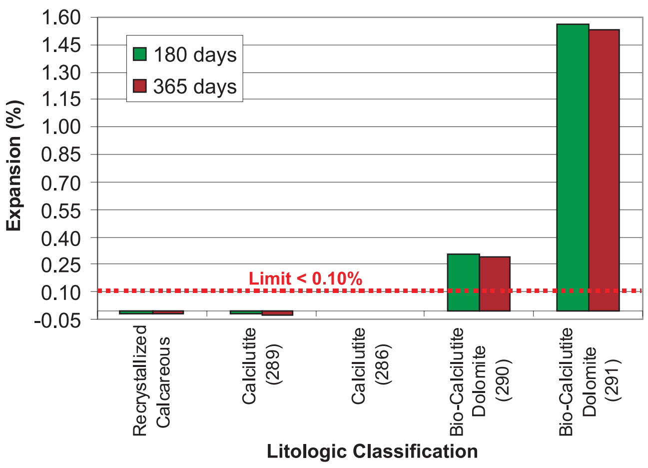

**Figure 4 – Microanalysis of gel from Figure 3**




### 3.2.2 ACR Investigation – Rock-Cylinder Method - NBR 10340 [8]/ASTM C-586 [9]

The expansion results of cores from 5 rocks evaluated by NBR 10340 [8] are presented in Figure 5, by the age of 180 and 365 days.
According to ASTM C-586 [9], expansion up to 0.10% is an indicative of deleterious reaction due to ACR. NBR standard does not indicate limits.
Hence, based on accomplished tests, it is certified the reactivity potential by ACR only in 2 rocks with bio-calcilutite dolomite lithotype. The remaining rocks did not present expansion rates. It is interesting to comment that these two rocks were the ones that also presented reactivity potential; however, this is due to ASR evaluated through ASTM C-1260 [7]. These two rocks hold mineralogical compositions with potential to develop any of the alkali-aggregate reactions, as characterization presented in item 3.1.
It is important to mention that a question always raised is about the representatively, once that a reduced sample from the rock is considered (35 mm of length and 9 mm of

Figure 5 – Potential reactivity for Carbonate Rock – Rock Cylinder Method

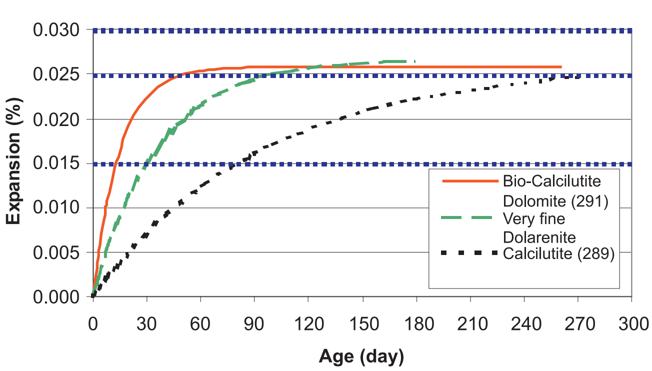



diameter) for being tested by this method. Even there are three samples for test, it could be difficult to guarantee the representativity of the population.

Analyses on the cores by DRX were performed. The comparison between the diffractrograms of rock in their natural state and the ones from mini-cores showed variations in the rock composition. The alterations in the mineral composition correspond to the disappearance of dolomite in the samples of dolomite bio-calcilutite rock (290 and 291). In the analyses made with core 291, the appearance of some reflections also suggests brucite formation. Thus, the analyses by DRX are important for the identification of the present stages, as well as of already existing minerals, assisting in the rock petrographic characterization and in the neoformed crystalline products from the deleterious reaction [12].

It is important to mention studies made by Tong & Tang [15], which indicate that the ACR degree can be monitored by the amount of consumed dolomite and production of calcite in cylinders with dolomite and by the amount of magnesite and brucite formed in the cylinders with magnesite. Although it is preferable to adopt the changes of the dolomite and magnesite through the monitoring of reaction degree in the cylinders, the identification of the reaction products calcite and brucite have proved to be as good as such. The factor of the amount of calcite to being higher from the one foreseen by dedolomite, suggests that the cement hydration products (e.g. portlandite) were reacted by the $CO_3^{2-}$ produced in the dedolomite. This reaction is known as concrete carbonization, however induced by the dedolomite product.

Figure 6 – Referring graph to the assay of Potential reactivity – ASTM C 1105 (10)




### 3.2.3 ACR Investigation – Concrete Prisms – ASTM C-1105 [10]

Figure 6 shows the expansive behavior of 3 carbonate rocks evaluated by the ASTM C-1105 method, in which exponential trend is observed.

According to ASTM C 1105 [10], expansions equal or superior to 0.015% by 3 months, 0.025% by 6 months or 0.030% by 1 year are indicative of a deleterious behavior of the cement/aggregate combination used; the expansion can be known as potentially deleterious.

It is observed for the three rocks evaluated that along the 90 days of age the expansions already surpass the 0.015% limit, showing, still, growth from this age and possibility of ACR development. However, it was not possible to register readings in more advanced ages due to the extensometers, used to measure length variation, and installed internally in the concrete did not accused readings, probably due to the commitment of its integrity in virtue of high observed expansions. Tests with steel studs should be done so they give continuity to the observation of the expansive behavior of these rocks.

This method (ASTM C1105 [10]) proves the expansions by ACR previously observed through the cores method for the bio-calcilutite type rock. The calcilutite rock investigated by concrete prisms method shows reactivity potential by the ACR, although it did not mean reactivity by the rock core method, showing incompatibility between the interpretation of the methodologies used for ACR.

The petrographic analyses [12] carried out after the tests with concrete prisms showed three alterations: 1) pellicles cutting the aggregate and the mortar, 2) associated to the presence of these pellicles, reaction edges occur, mainly in the aggregate, showed through the difference of coloration by microscopy optics and 3) pores partially filled (Figures 7 and 8). Its joint occurrence shows indications of the AAR development.

Analyses of some fragments removed from the concrete prisms, after reactivity test, were performed through scanning electron microscope (SEM) and EDX in samples with fracture surface. The results confirm ACR with the identification of some neoformed products, as shown at Figures 9 and 10. To confirm the identification of these products the DRX was used [12].

Figure 7

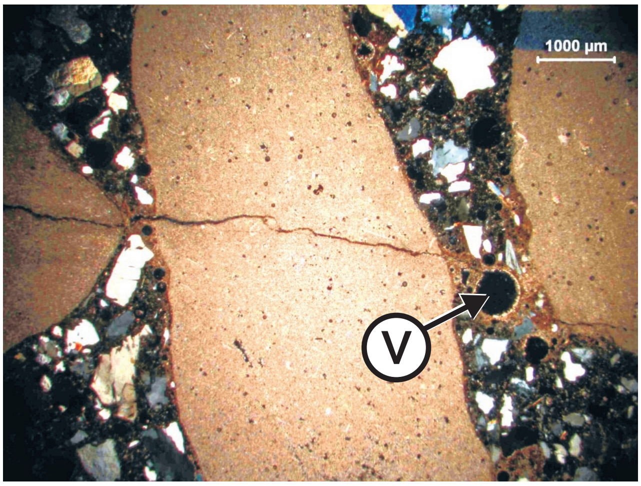
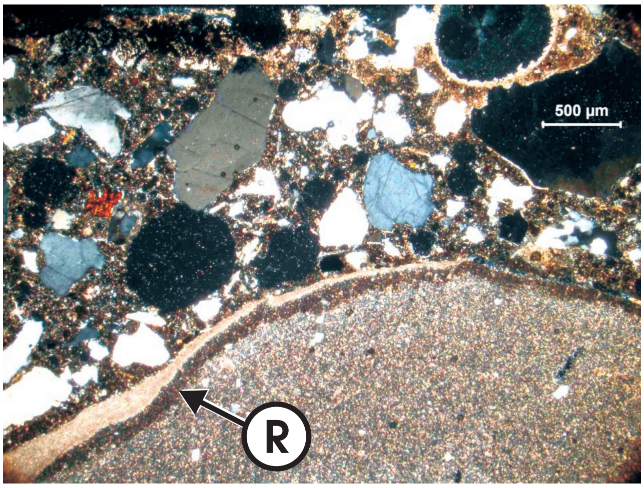



Figure 8

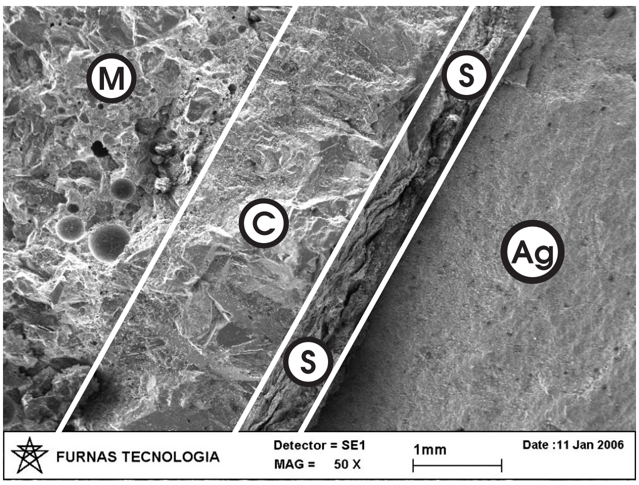



**Optical microscope photomicrographs of the more reactive concrete prisms (291). Cracking along mortar and aggregate with some material, voids filled (V) and reaction rims (R) around aggregate seen due to differences in color.**

### 3.2.4 ACR Analysis – Canadian Chemical Method – AAR 23.26-A [11]

The potentiality of the rocks for ACR that relates the ration CaO/MgO versus $Al_2O_3$, proposed by Roger [13] , and regulated in AAR 23.26-A [11] (Figure 11), shows the distribution of the studied samples according to its chemical composition. From the graph it can be seen that, from the 6 samples of rock, 3 of them show potential expansion by the chemical method, through results of CaO, MgO and $Al_2O_3$.
It is observed that these 3 rocks (286, 290 and 291) that behaved as reactive by the ASTM C-1260 [7], 2 by the ASTM C-586 [9] (290 and 291) and one (291) of the 3 reactive rocks investigated by ASTM C-1105 [10], are placed in a field of potentially reactive by the Canadian chemical method. That is, it is observed in this study, and with the analyzed samples, that there was incompatibility of behavior between chemical method and ASTM C-1105 [10] had. The remaining methods showed coherence between the final results.

Figure 9 – SEM micrograph of sample 289P. Film of fibrous material (S) followed by massive material (C) between mortar (M) and aggregate (Ag.)




### 3.2.5 Global Analysis from the Tests Methods

In Table 3 it is found a summary of the rocks behavior in relation to AAR, either ASR or ACR type from the investigated test methods.
It is observed that the rocks with bio-calcilutite dolomite lithotype present reactivity potential of type ASR as ACR, being confirmed by all the investigation methods.
Yet, one of the investigated calcilutite (286) shows ASR development by ASTM C-1260 [7] method and of ACR by

Figure 10 – Microanalysis of fibrous material presented in Figure 9

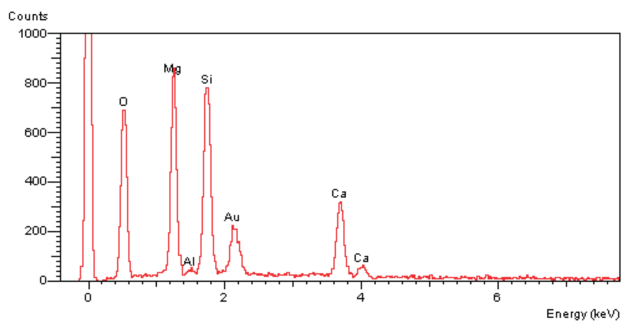
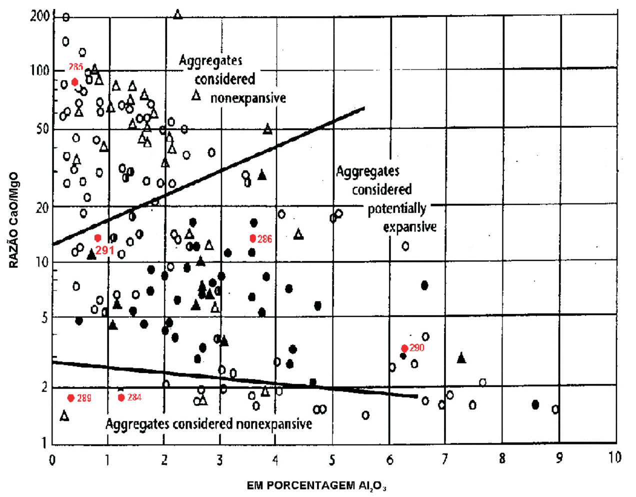


Figure 11 – Graph of CaO:MgO ration versus $Al_2O_3$ used by AAR 23.26-A (11), with results from the studied rocks




the chemical method while that the other calcilutite (289) accuses only ACR development, and only by a methodology (ASTM C-1105 [10]), in the same way that it occurs for the fine dolarenite (284). Yet, the re-crystallized calcareous does not show reactive potentiality by none of the employed methods.

Thus, it is verified that there are some incompatibilities among the ACR investigation methods, once that for some rocks (284 and 289) the ASTM C-586 [9] and Canadian method indicates innocuous behavior and the ASTM C-1105 [10], reactive behavior.

## 4 Conclusions

The conclusions presented here mention the results of analyses and tests performed in the studied samples.

The Alkali-Aggregate Reaction continues revealing itself to be quite complex, mainly when it is related to carbonate rocks, once that they can develop all types of existing reaction (alkali-silica, alkali-silicate, and/or alkali carbonate), depending on the rock mineralogy, being an individual or not.

Based on results, it can be elucidated that the application of only one methodology in the rock evaluation can result on not quite reliable results as observed in this study, once that the verified behaviors are not always coherent and compatible.

Thus, in general, it is verified the importance to investigate all available methodologies, once that the improper use of a rock, from the conclusion of only one test method, can supply a false-negative result and cause several problems to the concrete structure in the future. Additional investigations, and with other types of carbonate rocks must also be performed with the methods used in this study to confirm these hypotheses

Once the rocks did not show systematic results, it is also necessary the accomplishment of a detailed mineralogical, textural, and structural analysis after the performance of the test methods in order to confirm the AAR presence.

## 5 Aknowledgments

The present study is part of the research and development project of Aneel (P&D) entitled "Viability of Carbonate Aggregates Use in Dams", coordinated and managed by the Department of Support and Technical Control of Furnas Centrais Elétricas S.A., approved for the 2001/2002 cycle by the National Agency of Electric Energy - ANEEL. Therefore, the team thanks FURNAS CENTRAIS ELÉTRICAS S.A and ANEEL, in first place, for making possible this study accomplishment, as well as its divulgation. The team is also thankful all that have worked directly or indirectly in this project, as well as all technicians that performed these tests, in particular Dorival Tizzo, Renato Batista, Fernanda Mendonça, Alvaro Donizete, Aristides, the geologists Heloisa Helena B. Da Silva, Maria Celene Barbosa Ferreira and Antonio Sales Teixeira; and the researcher

Table 3 – Conclusive table on accomplished AAR assays

| Litologic Classification | Sample | Type of Investigation | | | |
|---|---|---|---|---|---|
| | | ASR | ACR | | |
| | | ASTM C-1260 | ASTM C-586 | ASTM C-1105² | AAR-23-26A |
| Very Fine Dolarenite | 284 | I | – | R | I |
| Recrystallized Calcareous | 285 | I | I | – | I |
| Calcilutite | 286 | R | I | – | R |
| Calcilutite | 289 | I | I | R | I |
| Bio-Calcilutite Dolomite | 290 | R | R | – | R |
| Bio-Calcilutite Dolomite | 291 | R | R | R | R |

**Obs. 1** - R - Reactive; I - Innocuous.
**Obs. 2** - The reactivity by ACR was observed to the 90 days, once that it surpassed the 0.015% limit. Some rocks had not been evaluated by the ASTM C-1105 (10).

Elcio Antonio Guerra, who contributed considerably to the experimental program.

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