Critical evaluation of test methods and experimental standards and non-standard on the alkali carbonate reaction in concrete

Chapter 1
INTRODUCTION: 1.0

The most common reaction of alkali aggregate reaction is alkali-silica reaction (ASR), but there is other kind of alkali aggregate reactions which has been recognized as being different from that of the more common and that is alkali-carbonate reaction (ACR) [1]. Alkali Carbonate Reaction (ACR) was first recognized in the late
1950s after the initial publication on alkali-reactive carbonate rock at Kingston, Ontario [4]. Alkali-Carbonate reaction (ACR) is defined as a mysterious reaction between the alkalis in Portland cement and any carbonate aggregate [1].

ACR reaction occurs with fairly select carbonate rocks, notably fine grained dolomites and fine-grained dolomitic limestone, usually with significant insoluble residue content. The reaction is more complex than ASR, involving chemical breakdown of dolomite (Dedolomitization) (Dolomite is the common name for calcium-magnesium carbonate) in the stone and formation of expansive reaction products [3].

"In the early 1990s, Katayama posited that ACR is a combined reaction of deleteriously expansive ASR of cryptocrystalline quartz, and harmless dedolomitization of dolomitic aggregate”[2].

Deleterious ACR, which causes progressive deterioration of concrete due to expanding cracks, and has been observed and studied by several authors in the past, is most likely a consequence of cryptocrystalline quartz, which is invisible in thin section microscopy, or clay content. Other forms of ACR – dedolomitization and the formation of reaction rims– should not be harmful to concrete. However, some carbonate rocks without silica content show significant shrinkage in accelerated laboratory tests”[].

Chapter 2
2.0 MECHANISM OF ACR

Dolomite rocks was observed to be mainly associated with alkali-carbonate reaction(ACR). Dolomite rocks known to be containing larger crystal od dolomite dispersed in and surrendered by a fine-grained matrix of calcite (calcium carbonate) and clay. ACR is somewhat scarce because aggregates oversensitive to the reaction are usually unsuitable for use in concrete [5]. Small amount of reactive silica can exist in the squashy dolomitic limestone which is contains calcite and dolomite. If the lithological characteristics were exist aggregates can be considering as potential for expansive ACR, and these characteristics are [5]:

• Clay content, or insoluble residue content, in the range of 5% to 25%
• Dolomite content (percentage in carbonate fraction) in the range of 40% to 60%.
• Interlocking dolomite grains (late expansion).
• Small size (25 to 30 μm), discrete dolomite crystals (rhombs) suspended in a clay matrix.

Chapter 3
3.0 TEST METHODS:
3.1 Petrographic Examination (ASTM C 295):

Petrographic examination is an important test since it gives clear definition about the character of rock. It is very easy to identify the potential alkali-carbonate aggregate by using petrographic test because these aggregates have lithology characteristics. Specimen rock can be considered reactive according to ACR if the texture consists of dolomitic rhombs float in a fine-grained matrix of calcite [5,6].

3.1.1. ADVANTAGES AND DISADVANTAGES:
ADVANTAGE:
• It gives quick results to help predict possible aggregate reactivity.
DISADVANTAGE:
• Can’t give quantitative information about the aggregate’s actual behavior in concrete.

3.2 Rock Cylinder Method (ASTM C 586):

This test Method is performed for Potential Alkali Reactivity of Carbonate Rocks for Concrete Aggregates. This method relayed on determining the expansive characteristics of carbonate rocks. The procedures of this test are first of all a small rock should be taken (cylindrical specimen) with specific dimensions of (35 mm specific long and 9 mm diameter). Secondly the specimen should be submerged in an alkaline (1 N NaOH) solution at room temperature. Finally, the specimen should be monitored for a year or less to observe the change of length for the specimen, there were one exception because the expansion influence usually appears after about 1 month which is that if the expansion of A 28-day reached 0.10% or more will reference to potential for deleterious expansion in a service environment [5].

3.2.1. Equipment:
• Length comparator.

3.2.2 advantage and disadvantage:
Advantage:
• The procedures of doing the test are easy.
Disadvantages:
• Length change of the specimen maybe monitored for up to or over a year.
• Obtaining a representative sample may be difficult.
• This test is intended only as a supplement to other test procedures.

3.3 Concrete Prism Tests (ASTM C 1105 and CSA A23.2-14A):
The purpose of this test is to measure the change of length in concrete due to alkali-carbonate rock reaction.
Six concrete prism specimen was used with an aggregate in question and the job cement. The duration of
this test is preferable for one year. In case test takes longer time is not functional. six or three months can
be appropriate. The expansion at one year should be equal or more than 0.03%,0.025 at six months, or 0.015 at
three months denote potentially deleterious aggregate [5].

prism should be stored over water in sealed containers at 100oF (38oC) and the change of length should be
under observation regularly. If the expansion of the prisms does not exceed 0.04 0r more after one year the
cement is considered to be non-deleteriously reactive and it can be use in cement with no need for further
testing. If the expansion of the prism exceeds 0.03% or more at 1 year, the aggregate is considered to be
potentially reactive and preventive measures are required. [7].

3.3.1. Equipment:
• Length comparator.
• Containers.

3.3.2. Advantages and disadvantages:
Advantage:
• This test is more beneficial to verify of the potential reactivity of an aggregate containing ACR-susceptible
rock.
• The test can be taken for six and three months.

Disadvantages:
• The test can be taken for one year.

3.4. Determination of Potential Alkali-Carbonate Reactive Rocks by Chemical Composition (CSA A23.2-26A):
The exist of this test was depending on the experience of Canadian. The mechanism of this method is by
identify the minerals of carbonate by using thin section analysis on bulk sample. The main required elements
to be analyzed are calcium, magnesium and aluminum. To be the reactivity estimated calcium-oxide
/magnesium-oxide ratio should be calculated and the calculations should be plotted in the chart against the
aluminum-oxide content. There will be to possibilities of results, which are considering potentially expansive
or considering non-expansive. In case the specimen was considered as potentially expansive ACR is probable
and further tests should be done. But if the specimen considered as non-expansive ACR is unlikely and there
is possibility for ASR, and test according to ASR should be done [7]

3.4.1. Equipment:
• X-Ray Fluorescence equipment.

3.4.2. Advantages and disadvantages:
Advantages:
• Get the results immediately.
• ACR is highly affected by the mineralogical composition of the rock and therefore XRF analysis can give
ear early vital information on ACR Expansion.

Chapter 4

4.0 COMPARISON BETWEEN THE TEST METHODS:
4.1 Comparison between 150°C autoclave method and concrete prism test:
CONCRETE PRISM TEST Determination of Potential Alkali-
Carbonate Reactive Rocks by Chemical
Composition
1-Can be used for assessed the potential of ASR and ACR. 1-Can be used to assessed the potential of ACR.
2- The results are available after one year or six months or three months. 2- It shows the results immediately.
3- Standard method. 3- Standard method.
4- If the prisms do not expand by more than 0.04% after 1 year, the aggregate is considered non-
deleteriously reactive and may be used in concrete with no further testing.
5- If the prism expands by more than 0.03% at 1 year, the aggregate is considered to be potentially reactive and preventive measures are required. 4- The specimen should be analyzed to identify the Chemical compositions.
5- If the results were plotted at the top on the chart it considers to be non-reaction but if it was plotted in the middle it considers potentially expansive, but if the results were plotted at the end of the chart it considered non-expansive.
4.2 Comparison between concrete prism test and rock cylinder method:
CONCRETE PRISM TEST ROCK CYLINDER METHOD
1-Can be used for assessed the potential of ASR and ACR. 1-Can be used to assessed the potential of ACR.
2- The results are available after one year or six months or three months. 2- Length change of the specimen may be monitored for up to or over a year.
3- Stander method. 3-Stander method.
4- If the prisms do not expand by more than 0.04% after 1 year, the aggregate is con~sidered non-deleteriously reactive and may be used in concrete with no further testing. 4- If the expansion of the specimen is less than 0.10% at 28 days the rock cylinder test should be continue for one year.
5- If the prism expands by more than 0.03% at 1 year, the aggregate is considered to be potentially reactive and preventive measures are required. 5- If the specimen expands after 28 days by 0.10% or more the specimen considered to be reactive and should be yield to prism test according to ACR (ASTM 1105).
Chapter 5
5.1. The effect of lithium nitrate on the course of the alkali-carbonate reaction:
This experimental study was carried out by Z. Owsiak and J. Zapala-Slaweta. The compounds of the lithium can help to initiated the alkali silica reaction from alkali carbonate reaction the results that were showed the inducted investigation that introducing lithium ions balanced by hydroxyl ions (in the form of fluoride, hydroxide or lithium carbonate) leads to increasing expansion level. Alkali hydroxides, causing the process of dedolomitization, lead to the formation of brucite, calcium carbonate and the carbonate of an alkali element. Nine mortar samples were prepared in this investigation. The alkali levels were 0.66, 0.9, and 1.1 of sodium equivalent for three samples and the appropriate alkali was reached by adding potassium sulfate into the mortar mix. The W/C ratio was 0.47 while the nitrate (Li/Na +k) percentage were 0.74. The sample expansion investigation was conducted through a lengthened period of 30 days. A1, C2 and A3, C3 the first samples were submerged in 1M NAOH solution at the temperature of 80c nevertheless the other group was submerged in NAOH and LIN03 solution at the temperature of 80c. petrographic and x-ray analysis for mortar samples with nitrate have been done. Was observed that the samples with alkali content of 0.9 % showed the highest expansion values. Large number of crack and spall was showed on the samples that modified with lithium nitrate. The conducted X-ray analysis of the spalls revealed the presence of small amounts of magnesium hydroxide and ettringite, and considerable amounts of quartz and calcspar. In sample modified of lithium nitrate one was observed that amorphous alkali silica gel is rich in calcium and sodium. In this samples (A3, C3) which were actually submerged in sodium solution and modified with lithium nitrate expansion did not occur because the lithium ions were being converted into silica gel and that decreased the expansive properties, but there were spalls on their surface and smaller number of fine cracks [10].
5.2. Influence of alkali carbonate reaction on compressive strength of mortars with air lime binder:
This experiential study was carried out by P. Stukovnik, M. Marinsek, B. Mirtic and V. Bokan Bosiljkov. Two mixtures of mortar were prepared (L mortar) consist of limestone and (D mortar) consist of dolomite aggregate. The dimension of the mortar bars is (40 ×40×160mm) according to SIST standard. Three bars were put on supporting system allowing uniform access of air to all surface of mortar bars. After that the bars were left in the laboratory conditions (temperature of 20c±3c and relative humidity of 60c ±5c%) for three months and later on the mortar bars were exposed to the different curing conditions to accelerate the ACR which are 1M NaOH solution at 20c, 1M NaOH solution at 60c and distilled in water at 60c. should to be optioned that ACR rate depended on PH and temperature of the solution. In the test method of change of length all specimens indicate shrinkage at 20c and 60c the shrinkage was lower when the mortar bar was exposed to aqueous environment than at the exposure at NaOHeq solution. All the mortar bars were exposed to the NaOH solution have showed higher shrinkage which implying that NaOH conditions accelerate lime mortar degradation. The compressive test results that was measured at the 0 time was 1.87 MPa and 1.80 MPa for the L- and D-sample but all L-samples start reducing to lower values after 1 month, 3 months, and 6 months by 1 MPa and 0.7 MPa respectively due to spreading of the applied solution through the porous system of the mortar and consequent dissolution and leaching of the lime binder. Compressive strength was increased in lime mortars prepared using the dolomite aggregate and it scored 2.2 MPa, 3.2 MPa and 4.6 MPa after 1, 3 and 6 months respectively. The observed effect can be attributed to the ACR, which progresses faster in the NaOHeq environment [9].

5.3. Alkali-carbonate reaction in concrete and its implications for a high rate of long-term compressive strength increase:

This experimental study was carried out by P. Štukovnik, T. Princ’ic, R.S. Pejovnik, V. Bokan Bosiljkov. The material that was used aggregate, two different aggregate were used one is limestone and the other is dolomite noted as A and B. Two different mixture were prepared (A mixture) contained limestone aggregate and (B mixture) contained dolomite rock. The binder used was a CEM I 42.5R Cement with very high level of early and final strengths. The water-to-cement (W/C) ratio and the aggregate-to-cement ratio of both mortars were 0.45 and 3, respectively. The mortar bars were casted with dimensions of (60×60×160mm3). After that they were cured in an environment of relative humidity of 90% and temperature of 20ºc for 28 days. After that all specimen at the age of 28 days were exposed to four exposure conditions: distilled water at 20ºC, 1 M NaOH solution at 20ºC, distilled water at 60ºC and 1 M NaOH solution at 60ºc. The microscopic tests and the mechanical tests were carried out after 0, 3 ,6 and 12 months.

The tests methods that were used are microscopic observation with two different optical microscopes were used, which are 3D microscope system HIROX KH 3000 and NIXON Eclipse E 200, a FE-SEM Zeiss Ultra Plus microscope equipped with EDS to detect the process of dedolomitization, and X-ray diffraction analysis was used for the mineralogical detection of carbonate rocks.

The result that was observed on the compressive and flexural strength for the specimen (A-H20-20C) is increased by 13% between the value at 0 months by 46 MPa and the value after exposure to water at 3 months by 52 MPa. To extend the exposure (up to 6 months) to the same condition did not increase compressive strength any further. However, when the specimen A was exposed to the accelerated solution significant increase was observed between 3 months and 6 months by 10 MPa. We can conclude that the alkalinity of the solution used influences the rate of increase in compressive strength over time for Mixture A at 20 ºC. On the other hand, when the exposure temperature was 60 for the specimen (A-H20-60C) at 6 months and (A-NH-60C) at 6 months were 4% and 30% lower compared to the specimens (A-H20-20C) at 6 months and (A-NH-20C) at 6 months respectively, that means high temperature and high alkalinity effect positively on the compressive strength on the specimen A. In the case of the specimen B the average compressive strength is considerably higher than for Mixture A under all four exposure conditions. Exposure to 20ºC distilled water for 3 and 6 months resulted in 22% and 46% higher compressive strength in Mixture B than in Mixture A, and at 60ºC the increase was 41% after 6 months. Exposure to the NaOH solution increased differences in compressive strength between the two mixtures even further, by 35% after 3 months at 20ºC and 88% after 6 months at 60ºC. A lower increase (25%) was observed in NaOH solution after 6 months at 20ºC. The difference in the strength increase between mixture A and mixture B for flexural strength is considerably lower than for compressive strength, and it seems that exposure to the accelerated NaOH solution does not influence the results for flexural strength, at least not in a positive way, as is the case for
compressive strength.

In the petrographic analysis for aggregate A showed that the rock is biomicrite limestone with high homogeneity. Crocks are rare, very small and filled with calcite sparite cement. X-ray analysis showed that aggregate A composed of mineral calcite 99% and mineral dolomite 1%. In the petrographic analysis for aggregate B shows that it is late diagenetic Dolomite. The pre-cracks are fill with calcite and dolomite sparite cement. The result of the x-ray analysis showed that rock containing dolomite presence 97% and calcite presence 3%. Petrographic analysis after exposure to 1M NaOH solution for 9 months. Limestone bars stayed unchanged and inert, but in the dolomite rock samples the process of dedolomitisation was detected, usually along pre-existing cracks. It is evidenced by recrystallization, and a change in the color of the dolomite crystals from white to dark brown. Also, was observed a regarding change in the porosity of the rock, pores are more frequent and bigger than the reference samples, that was due to the recrystallization of sparite dolomite crystal and to the smaller volume reaction products. And the increase of porosity was due to the equation of dedolomitization with a volume change of 1.5% [11].

Conclusion:

References:
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