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Effect of Clay Content on Alkali-Carbonate Reactive (ACR) Dolomitic Limestone

by

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13. Abstract

This study aimed to evaluate whether an aggregate's clay content plays an overarching role in ACR expansion and deterioration within the limestone sources that have been approved for use in portland cement concrete in Louisiana. A total of 29 aggregate sources from the Louisiana Department of Transportation and Development (DOTD) approved materials list were screened for chemical analysis to determine whether the aggregates were potentially expansive based on their calcium oxide, magnesium oxide, and alumina contents. Twelve out of 29 aggregates were found to be potentially reactive and therefore were selected for concrete prism tests per ASTM C1105 to verify their expansive potential. The results showed that none of the aggregates tested exhibited deleterious expansion after 12 months. In addition, the clay content (based on the aggregate's alumina composition) did not directly affect the selected group of aggregates' reactivity.

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Abstract

This study aimed to evaluate whether an aggregate's clay content plays an overarching role in ACR expansion and deterioration within the limestone sources that have been approved for use in portland cement concrete in Louisiana. A total of 29 aggregate sources from the Louisiana Department of Transportation and Development (DOTD) approved materials list were screened for chemical analysis to determine whether the aggregates were potentially expansive based on their calcium oxide, magnesium oxide, and alumina contents. Twelve out of 29 aggregates were found to be potentially reactive and therefore were selected for concrete prism tests per ASTM C1105 to verify their expansive potential. The results showed that none of the aggregates tested exhibited deleterious expansion after 12 months. In addition, the clay content (based on the aggregate's alumina composition) did not directly affect the selected group of aggregates' reactivity.

Acknowledgments

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Implementation Statement

The results from this study provided insight into whether a dolomitic limestone's clay content affected concrete deterioration through alkali carbonate reactivity (ACR). In addition, the results from the ACR testing (through ASTM C1105) were used to determine whether any limestone aggregates should not be allowed for use in portland cement concrete by DOTD due to excessive expansion and to protect DOTD against ACR deterioration.

Table of Contents

Technical Report Standard Page	1
Project Review Committee	2
LTRC Administrator	2
Members	2
Directorate Implementation Sponsor	2
Effect of Clay Content on Alkali-Carbonate Reactive (ACR) Dolomitic Limestone.....	3
Abstract	4
Acknowledgments.....	5
Implementation Statement	6
Table of Contents	7
List of Tables.....	8
List of Figures.....	9
Introduction.....	10
Literature Review.....	11
Mechanism of ACR.....	11
Factors that Contribute to ACR Expansion.....	13
Characterization Methods of ACR.....	14
Mitigation Strategies.....	17
Objective.....	20
Scope.....	21
Methodology.....	22
Coarse Aggregate Screening.....	22
Concrete Testing	22
Concrete Mixture Design.....	23
Discussion of Results.....	24
Chemical Analysis	24
Fresh Concrete Properties.....	25
Length Change Results	26
Relationship Between ACR Expansion and Clay Content	27
Conclusions.....	29
Recommendations.....	30
Acronyms, Abbreviations, and Symbols.....	31
References.....	32

List of Tables

Table 1. Criteria for evaluating ACR expansion (ASTM C1105).....	22
Table 2. Description of coarse aggregate sources selected for the concrete prism tests...	25
Table 3. Fresh concrete properties results	26
Table 4. Concrete prism test results	27

List of Figures

Figure 1. Morphology of a typical carbonate rock susceptible to ACR [29]	14
Figure 2. A plot of CaO/MgO ratio versus the Al ₂ O ₃ content of quarried carbonate rocks [48].....	16
Figure 3. Chemical analysis results	24
Figure 4. Relationship between ACR expansion and clay content	28

Introduction

Alkali-aggregate reactivity (AAR) can cause significant damage in concrete due to an internal swelling reaction from reactive aggregates. This distress mechanism is manifested in the form of alkali-silica reaction (ASR) or alkali-carbonate reaction (ACR). In ASR, aggregates with silicate or silica minerals react with alkaline solutions found in cement to create an alkali-silicate gel that, in the presence of moisture, causes expansion and cracking of the affected aggregate particles and the surrounding cementitious matrix [1]. In ACR, its expansion mechanism is theorized to be triggered by the dedolomitization of carbonate rocks [2]. This process occurs when a dolomite crystal combines with the alkalis in solution found in portland cement to form brucite, potassium, and calcium carbonates. The subsequent crystallization of brucite is said to cause significant expansion within concrete, resulting in considerable deterioration and cracking [3] [4].

In North America, ACR was first documented by Swenson [5] of the National Research Council of Canada. General indicators of ACR expansion include a map or pattern cracking, closed joints, blow-ups or crushed concrete, and relative offsets of adjacent slabs or substructures [4] [6]. Pavements containing alkali-carbonate reactive aggregates have been found in Louisiana on Interstate 20 between Ruston and Monroe and also near the Mississippi River bridge [7]. Other paving and structural projects in Louisiana have also been identified as having ACR-susceptible aggregates. Because of these investigations, DOTD specifications were updated to reduce the risk of ACR deterioration in concrete using AASHTO PP65-11 [8] [9].

Aggregates can be prone to ACR expansion if (1) the clay content or insoluble residue content is in the range of 5-25%; (2) the dolomite content is in the range of 40-60%; (3) there are interlocking dolomite grains; and (4) if small discrete dolomite crystals (25-30 microns) are suspended in a clay matrix [10] [11] [12]. It is worth noting that ACR can also be triggered without a dedolomitization reaction, where the expansion is driven by chemical reactions between the clay minerals in the aggregate matrix instead [7].

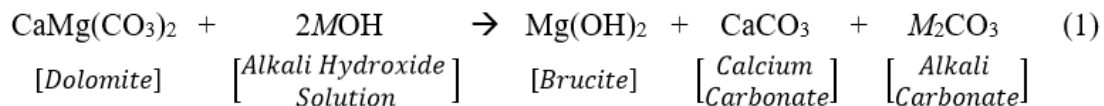
However, it is unclear to what extent clay content contributes to ACR, particularly for aggregates that are suitable for concrete construction. For this reason, this study aims to evaluate whether an aggregate's clay content plays an overarching role in ACR expansion and deterioration within the limestone sources that have been approved for use in portland cement concrete in Louisiana.

Literature Review

Mechanism of ACR

Alkali-carbonate reaction (ACR) is a deleterious reaction that results in the expansion and cracking of concrete in the presence of moisture, leading to premature deterioration and reduced service life. To this date, there is no consensus within the literature on what exactly triggers ACR in concrete. Rather, there are five theories that describe the mechanism behind ACR as summarized by Beyene et al. [13]: (a) expansion through dedolomitization [3] [5] [14] [15] [16] [17] [18] [19] [20]; (b) non-expansive dedolomitization in conjunction with the expansion caused by swelling of clay minerals from the uptake of water [21] [22] [23]; (c) alkali-silica reaction (ASR) misdiagnosed as ACR [24] [25] [26] [27] [28] [29]; (d) expansion through a combination of ASR and clays [30]; and (e) volumetric instability of dolomites caused by a substitution of iron for magnesium in its crystalline structure [31] [32].

Dedolomitization refers to the breaking down or dissolution of dolomite. Since dolomite is unstable in high pH environments, it can be partially dissolved when exposed to the alkali hydroxide solutions in concrete, increasing the aggregate's porosity [33] [34]. This reaction then leads to the formation of calcite and brucite, as described by equation (1), where M represents an alkali metal such as potassium, sodium, or lithium [2]. It has been hypothesized that ACR expansion is caused by brucite exerting pressure during crystallization [17]. The size and abundance of brucite is related to the reactivity of the dolomitic aggregate [35]. According to this theory, clays are not needed for ACR expansion to occur [4].

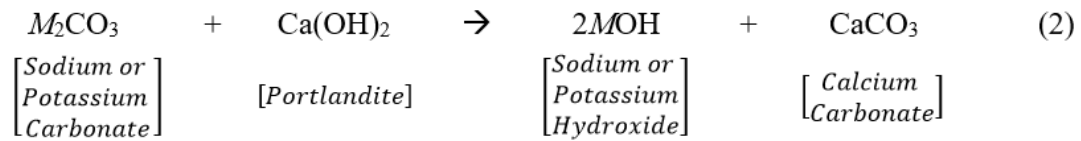


However, dedolomitization has also been characterized as a non-expansive reaction by several researchers, where the formation of brucite actually causes a volume reduction [24] [27] [34] [35]. These observations indicate that an additional reaction besides dedolomitization must be causing deleterious expansion [33]. Gillot and Swenson [23] reported that dedolomitization increases the aggregate's porosity just enough to allow alkalis and moisture in the aggregate's clay, which subsequently swells and causes deleterious expansion [21] [22]. Xu et al. [36] linked ACR to the aggregate's clay content

and dolomite crystal structure. Wong [7] suggested that ACR expansion found in a Louisiana highway was caused by a reaction between the alkalis in the cementitious matrix and the aggregate's clay minerals. Stokowski and Sarson [37] argued that clay minerals are fundamental to initiate ACR expansion, while Hadley [2] contended that clays must include reactive silica for expansion to take place.

Alternatively, Katayama [27] proposed that ACR is the combination of an expansive alkali-silica reaction of cryptocrystalline quartz and a non-expansive dedolomitization reaction that forms brucite and carbonate. Several researchers have also argued that ACR expansion is either misdiagnosed as ASR (since cryptocrystalline quartz may be too small to be detected in polarizing light microscopy) [24] [25] [38] or caused by a combination of ASR and clays [30]. It is also possible that carbonate rocks such as siliceous limestones and siliceous dolostones are susceptible to both ASR and ACR [39].

While dedolomitization in itself does not seem to cause damaging expansion, it may facilitate the occurrence of ASR [27]. Since dedolomitization usually increases the aggregate's porosity, this can increase the exposure of any cryptocrystalline silica present in the aggregate to concrete's alkaline pore solution, which helps initiate ASR [27]. In addition, sodium and potassium carbonate formed during dedolomitization from equation (1) reacts with calcium hydroxide from the hydrated portland cement paste, in a process known as the regeneration of alkali hydroxides as described by equation (2), which can further contribute to the formation of ASR gel.



It is worth noting, however, that ASR mitigation strategies such as using low alkali cements and pozzolans are inadequate for suppressing ACR [40] [41] since ACR can still be triggered at low alkali levels [11]. According to Katayama [27], this may be attributed to a slower rate of pozzolanic reactions relative to ASR with fast-reacting silica and the high alkalinity and pH maintained by the dedolomitization process.

Researchers have also noted cases where dedolomitization was not required to initiate ACR expansion. Mather et al. [42] and Buck [43] reported ACR in coarse limestone aggregates with no dolomite. Wong [7] reported deleterious ACR expansion in concrete with dolomitic limestones that showed no evidence of dedolomitization, as there was no brucite formation or dolomite alteration observed. Given the complex nature of ACR

expansion, there is still no consensus within the literature that accurately explains the mechanism of ACR.

Factors that Contribute to ACR Expansion

Aggregate Size

The nominal maximum size of the reactive aggregate influences the amount and extent of ACR, where a smaller maximum aggregate size results in a lower degree of expansion [44] [45].

Pore Solution Alkalinity

Aggregates that are susceptible to ACR are affected by the cement paste's pore solution alkalinity. As the pH of the pore solution increases, the likelihood for ACR increases, mainly because dolomites are increasingly unstable in alkaline environments [33]. During the dedolomitization reaction, the calcium hydroxide produced by portland cement hydration can react with the alkali carbonate produced from equation (1) to form an alkali hydroxide and calcium carbonate as described by equation (2). This reaction regenerates alkalis, reduces the concentration of carbonate ions, and exacerbates the dedolomitization reaction. For this reason, ACR can still occur in low-alkali cements and concretes with supplementary cementitious materials [41].

Aggregate Mineralogy

Carbonate rocks that are prone to ACR are typically defined by the presence of rhombic-shaped dolomite crystals ($\text{CaMg}(\text{CO}_3)_2$) in a fine-grained calcite matrix (CaCO_3), clay, and quartz [4]. These rocks tend to have a dilute hydrochloric acid-insoluble residue that contains a significant amount of clay [46]. Figure 1 shows a microscopic image of an ACR susceptible aggregate featuring a fine-grained, argillaceous matrix with small rhombic dolomite crystals [29].

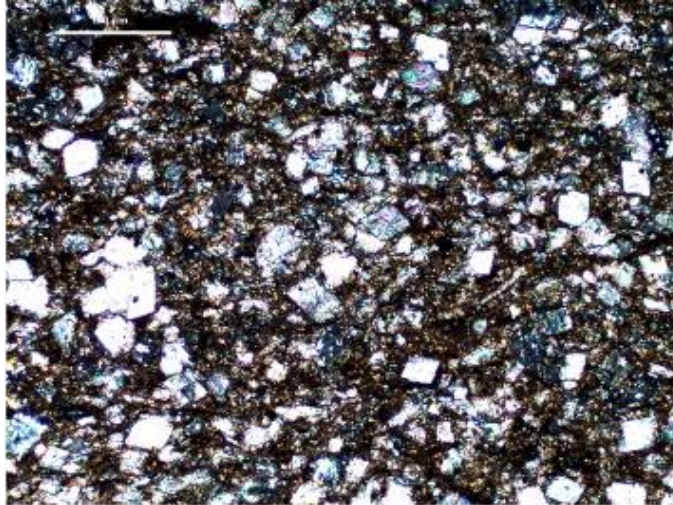


Figure 1. Morphology of a typical carbonate rock susceptible to ASR [29]

Ozol [12] and Swenson and Gillot [10] noted that aggregates could be prone to ASR expansion if (1) the clay content or insoluble residue content is in the range of 5-25%; (2) the dolomite content is in the range of 40-60%; (3) there are interlocking dolomite grains; and (4) if small discrete dolomite crystals (25-30 microns) are suspended in a clay matrix.

Characterization Methods of ASR

ASTM C1778 and AASHTO PP65 have published guidance on the characterization and mitigation of ASR in concrete construction [9] [47]. While ASR is less common than ASR, each aggregate source must be thoroughly examined before use in concrete as it is difficult to control ASR once expansion has begun. ASTM C1778 recommends the following plan of action to determine an aggregate's reactivity: (1) The use of field performance history, (2) petrographic assessment, and (3) a determination of ASR potential through a chemical analysis per CSA A23.2-26A [48]. If the aggregate has been deemed potentially reactive, a concrete prism test for ASR determination can be used per ASTM C1105 [49].

Field Performance History

An aggregate's field performance history is invaluable to understand whether an aggregate is susceptible to ASR. For example, the following information from an aggregate's record of service can help determine whether an aggregate is suitable for new

construction: (a) the structure's age; (b) the alkali loading of the concrete; (c) the amount of SCMs used; (d) exposure conditions; and (e) whether any distresses were observed. The structure's age is critical, as the alkali-aggregate reaction can take years before any damage is apparent. For this reason, ASTM C1778 recommends that an aggregate's field performance should be surveyed on structures that are at least 15 years old [47].

If the exposure conditions and the concrete mixture design (including total alkali content, SCMs) will be virtually the same in the existing and proposed concrete structures, it is not necessary to test an aggregate that has a good record of service. However, the aggregate should be tested if at least one of the following conditions are met: (a) the aggregate's properties vary significantly within the quarry, (b) the exposure conditions of the proposed structure will be more severe than in the existing structure that exhibited good performance, and (c) the concrete mixture design is considerably different. If an aggregate's field performance history is not available, laboratory tests are needed to evaluate the potential reactivity of aggregate [11] [47].

Petrographic Assessment (ASTM C295/ASTM C856)

The standard guide for petrographic examination of aggregates for concrete (ASTM C295) outlines the techniques used to characterize alkali-carbonate reactive constituents [50]. Aggregates that are prone to ACR have a particular composition that can be readily identified through a petrographic analysis, as they are primarily calcareous dolomites or dolomitic limestones with clayey insoluble residues. The rock is considered to be potentially reactive if it constitutes a fine-grained matrix of calcite and clay surrounding rhombic crystals of dolomite [11]. However, there have been cases where ACR expansion was observed in aggregates that did not have the aforementioned features [51]. For this reason, petrographic examination is often supplemented with expansion testing to classify an aggregate's reactivity since some reactive phases may not be detected by optical microscopy. Nevertheless, if the aggregate in question comes from a well-known and tested source, petrography alone can be used to classify such aggregate's reactivity. Alternatively, if a concrete sample is suspected of having been damaged by an alkali-aggregate reaction, a petrographic examination of hardened concrete per ASTM C856 [52] can be used to determine whether ACR or ASR caused the expansion. In this case, the main features that distinguish ACR from ASR are the dedolomitization reaction products such as brucite, calcite, and magnesium silicates [4].

Chemical Analysis (CSA A23.2-26A)

Rogers [53] developed a procedure to screen potentially reactive rocks based on their chemical composition, which is now a standardized test method (CSA A23.2-26A). This test determines the lime (CaO), magnesia (MgO), and clay content (estimated by its alumina (Al_2O_3) composition) of the sampled rock and determines where the composition of the rock falls on a plot of CaO/MgO ratio versus the Al_2O_3 content, as shown in Figure 2. If the aggregate's CaO/MgO ratio falls in the range of "aggregates considered to be potentially expansive" in Figure 2, the aggregate should be tested per ASTM C1105 to verify its reactivity.

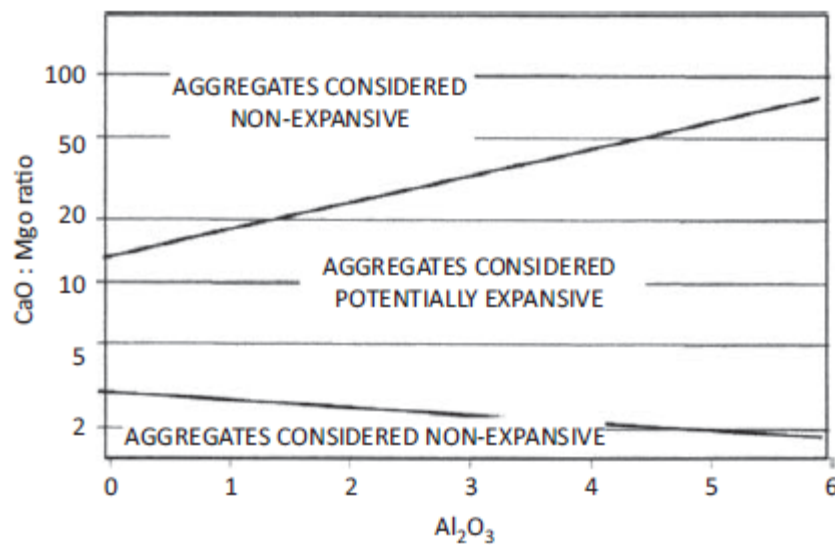


Figure 2. A plot of CaO/MgO ratio versus the Al_2O_3 content of quarried carbonate rocks [48]

Rock Cylinder Method (ASTM C586)

The test method for potential alkali reactivity of carbonate rocks for concrete aggregates, also known as the rock cylinder method [54], was developed by Hadley [15] to determine whether a rock will expand when immersed in an alkali solution (1 N NaOH) [4]. The test method provides a rapid indication of a rock's reactivity, where an aggregate is considered reactive if it exhibits an expansion greater than 0.10% at 28 days. However, some aggregates have been found to contract before they expand at later ages [55]. In such cases, an alternate expansion threshold of 0.2% at 16 weeks has been suggested by researchers [56] [57] [58].

This test method's main drawback is that it does not accurately predict the level of expansion it can cause in concrete since it does not consider the role of cement in ACR [12]. In addition, representative sampling can be challenging due to the inherent variability within rocks [12] [59]. For these reasons, ASTM C586 should not be used as an acceptance test [4]. Rather, those rocks that have been identified as expansive by ASTM C586 should also be tested in a concrete prism per ASTM C1105 to verify their expansion potential.

Concrete Prism Test (ASTM C1105)

The test method for length change of concrete due to alkali-carbonate rock reaction (ASTM C1105) is used to verify whether an aggregate is prone to ACR. Six concrete prisms measuring 285 mm long with a 75-mm square cross-section are prepared with the aggregate of interest (with a maximum size of 19 mm) and the job cement. The test should preferably be run for one year, but test results recorded at the 3-month or 6-month period can be acceptable if longer test times are not feasible. The suggested criteria for determining whether an aggregate is potentially expansive was based on works published by Newlon et al. [56] and Buck [60], where an expansion greater than or equal to 0.030% at one year, 0.025% at 6 months, or 0.015% at 3 months indicates a potentially deleterious aggregate.

This test determines whether a carbonate rock can be deleteriously expansive in concrete and takes a pragmatic approach by testing the proposed materials for an upcoming project. In addition, it is a more robust test than the rock cylinder method (ASTM C586) since some carbonate rocks found to be reactive by ASTM C586 may not necessarily contribute to deleterious expansion in concrete [44] [56]. However, the concrete prism test's 1-year duration makes it less practical for a routine aggregate inspection.

Mitigation Strategies

Selective Quarrying

If an aggregate source is deemed to be potentially reactive based on the rock cylinder test (ASTM C586), the best preventive measure is to avoid using such an aggregate by selective quarrying [61]. This is achieved by routinely mapping and testing quarries to define the rock groupings and stratification. Once identified, selective quarrying can be used to avoid the reactive strata [4] [11].

Blended Aggregate

ACR expansion is related to the proportion of reactive aggregate used [4]. Therefore, if an aggregate source is suspected to be susceptible to ACR, and if it is not economically feasible to avoid such reactive aggregate, aggregate dilution is recommended by blending the reactive aggregate with aggregates that have been proven to be nonreactive [59]. This is achieved by establishing a maximum of 20 % reactive coarse or fine aggregate, or using no more than 15% of a combined reactive coarse and fine aggregate [56] [62]. Under these limits, the blended aggregate may be used in portland cement concrete only if it tests satisfactorily in the concrete prism test (per ASTM C1105) without resulting in deleterious expansion.

Aggregate Size

Research has shown that a larger aggregate size can increase the degree of expansion in ACR-susceptible aggregates. As such, ACR expansion can be mitigated by limiting the aggregate's nominal maximum size to the smallest practical level [10] [59].

Moisture Availability

Since water is at least partly involved in triggering ACR, it is possible to limit the extent of ACR deterioration and prolong concrete's service life if the moisture exposure can be reduced [4].

Use of Pozzolans

Unlike ASR, ACR cannot be effectively controlled using pozzolans [4] [11] [44]. However, they may be able to slow down the rate of reactivity to a limited extent since pozzolans can reduce concrete's permeability and therefore limit the rate of migration of the alkali hydroxides [4]. At high replacement levels, deleterious ACR expansion was mitigated in a concrete prism test with 85% slag with the Pittsburg aggregate (a reference ACR aggregate) [27]. Therefore, while high amounts of SCMs could potentially mitigate ACR, these amounts are usually too high to be practical [45].

Cement Alkalinity

It has been well documented that low alkali cements are not effective in controlling deleterious ACR [2] [41] [44]. However, as a preventive measure, ACI recommends using cement with alkali levels lower than 0.4% [4].

Objective

The objective of this study was to determine whether an aggregate's clay content has an effect on alkali carbonate reactivity (ACR) and to evaluate whether DOTD's approved material list remains current and does not allow any aggregates that exhibit excessive ACR expansion.

Scope

The scope of this study was to evaluate the influence of a limestone's clay content on its alkali carbonate reactivity. This was achieved by screening all of the limestone sources that are included in DOTD's approved materials list for potential aggregate reactivity (per CSA A23.2-26A). If an aggregate was deemed to be potentially reactive, a standardized test method that measures the length change of concrete due to alkali-carbonate rock reaction (ASTM C1105) was used to characterize the aggregate's reactivity. The results of this study were limited to concrete mixtures that were designed similarly to the proportions described in ASTM C233.

Methodology

Coarse Aggregate Screening

An inventory of limestone aggregates in DOTD’s approved materials list (AML) was compiled for evaluation with the AASHTO PP 65 screening test, where 29 aggregates were screened. Specimens were prepared for analysis with X-Ray Fluorescence (XRF) equipment, where calcium oxide (CaO), magnesium oxide (MgO), and alumina (Al₂O₃) contents were determined in accordance with CSA-A23.2-26A. The clay content in aggregate can be estimated based on its alumina content.

The chemical analysis was also used to determine if the aggregates are considered potentially expansive based on a plot of CaO/MgO ratio versus Al₂O₃ contents. If the composition of the aggregate did not fall in the ‘potentially expansive’ range, the aggregate was considered to be unreactive and was not tested for the ASTM C1105 concrete prism test.

Concrete Testing

Once the aggregates were screened by chemical analysis, those classified as potentially expansive were tested in concrete prisms for length change to evaluate their susceptibility to ACR per ASTM C1105. These specimens were monitored for 12 months and were classified based on the limits described in Table 1. Fresh concrete properties such as slump (ASTM C143), air content (ASTM C231), and unit weight were also measured for all concrete samples.

Table 1. Criteria for evaluating ACR expansion (ASTM C1105)

Testing Age	Expansion %
12 months	0.030
6 months	0.025
3 months	0.015

Concrete Mixture Design

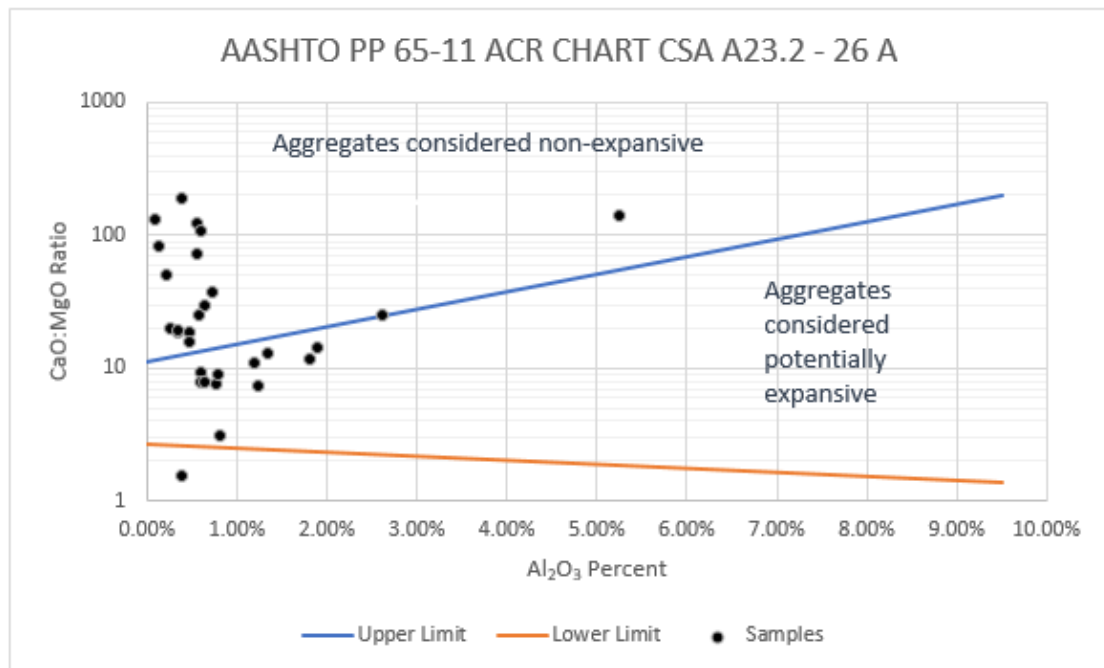
The concrete mixture design was initially based on ASTM C233 [63], which is the default mixture design called out by ASTM C1105 when no other specific concrete mixture is under consideration. ASTM C233 requires a fixed cement content at 517 lbs/yd³, with a targeted 3.5 in. \pm 0.5 in. slump and a 5.5% \pm 0.5% air content by varying the water content and the air-entraining dosage. However, given the inherent variability within each aggregate source and the difficulty of meeting the slump and air content targets, the mixture design was instead modified to resemble a more traditional DOTD concrete mixture design, with a fixed 0.45 w/c ratio and a 60/40 coarse-to-fine-aggregate ratio. These modifications should not have an effect on the ACR expansion as the factors that influence the degree of reactivity are the maximum nominal aggregate size and pore solution alkalinity. The Type I cement used for all samples in this study had an equivalent alkali content of 0.46%. All coarse aggregates featured a No. 57 gradation, and an air-entraining agent was used at 2 oz./cwt. Superplasticizers were added as needed to ensure workability, without exceeding 2 oz./cwt.

Discussion of Results

Chemical Analysis

A total of 29 aggregate sources were screened by a chemical analysis using x-ray fluorescence (XRF) per CSA A23.2-26A. The results were plotted in Figure 3, which indicates the region in which the aggregates are considered potentially expansive based on their calcium oxide/magnesium oxide ratio and alumina content. From these aggregates, 12 sources were in the region where aggregates are considered potentially expansive and therefore were selected to be tested for the concrete prism test per ASTM C1105.

Figure 3. Chemical analysis results



From the aggregate sources selected, their mineralogy ranged from dolomitic limestone, siliceous limestone, and oolitic stone. The details of the aggregates that were deemed to be potentially reactive were summarized in Table 2.

Table 2. Description of coarse aggregate sources selected for the concrete prism tests

Sample ID	Source	Aggregate Type
C4467	Kentucky	Siliceous Limestone
C4477	Kentucky	Siliceous Limestone
C4483	Kentucky	Siliceous Limestone
C4510	Missouri	Siliceous Limestone
C4533	Kentucky	Siliceous Limestone
C4573	Missouri	Oolitic Stone
C4608	Kentucky	Limestone
C4652	Texas	Limestone
C4705	Kentucky	Siliceous Limestone
C4706	Alabama	Limestone
C4707	Alabama	Limestone
C4709	Illinois	Siliceous Limestone

Fresh Concrete Properties

The slump, air content, and unit weight were measured for each specimen group, and the results were summarized in **Error! Reference source not found.** For the first specimen groups, a 0.57 water-cement (w/c) ratio resulted in high slumps and moderate air contents. In order to reduce the slump content closer to the targeted 3.5 in., the w/c was reduced to 0.45. Since this modification resulted in low slumps and moderate air contents, the mixture design was further modified to include a superplasticizer (dosed at 2 oz./cwt) to increase concrete's slump. The specimen groups ranging from C4573-C4652 closely approximated the 3.5-in. slump target, yet the air contents increased significantly due to the interactions between the air-entraining agent and the superplasticizer. The last mixture design modification made on specimens C4705-C4709 involved a slight reduction in superplasticizer to reduce the high air contents observed on the previous specimen groups.

With the exception of specimens C4533 and C4706, all slumps ranged from 2 in. to 5 in., which are satisfactory levels for structural and paving applications per DOTD specifications. Lower slumps (< 2 in.) are also acceptable for slip-form applications. With respect to air content, DOTD specifications recommend maintaining air within 4 – 7% by volume, a criterion that half of the specimen groups met. However, as previously noted,

these slight changes in the concrete mixture design (and thereby their corresponding fresh concrete properties) should have a negligible effect on ACR expansion.

Table 3. Fresh concrete properties results

Sample ID	Aggregate Type	Slump (in.)	Air Content (%)	Unit Weight (lb./ft³)	W/C Ratio
C4467	Siliceous Limestone	2.50	5.6	146.3	0.57
C4477	Siliceous Limestone	5.00	4.1	147.8	0.57
C4483	Siliceous Limestone	5.00	3.9	147.8	0.57
C4510	Siliceous Limestone	2.50	5.2	146.7	0.45
C4533	Siliceous Limestone	0.75	4.0	148.3	0.45
C4573	Oolitic Stone	2.25	5.6	143.0	0.45
C4608	Limestone	2.75	7.2	145.0	0.45
C4652	Limestone	3.50	10.0	135.9	0.45
C4705	Siliceous Limestone	5.00	9.0	141.2	0.45
C4706	Limestone	1.50	5.2	146.1	0.45
C4707	Limestone	2.50	7.8	145.0	0.45
C4709	Siliceous Limestone	2.00	7.4	144.3	0.45

Length Change Results

The 12-month length change of concrete due to ACR was measured for each specimen group per ASTM C1105 and summarized in Table 4. Using Table 1 to interpret the results, it can be observed that no specimen group exhibited deleterious expansion as all the length change percentages measured were lower than 0.030%. However, two sources from Kentucky were close to the 0.030% threshold, namely C4533 and C4483. In such borderline cases, consideration should be given to how the laboratory conditions compare with the proposed new construction's field conditions for acceptance, the costs associated with the aggregate choice, and how cracking may affect the proposed concrete's service life [4]. Nevertheless, the current results indicate that DOTD's approved materials list is adequate and does not need modification as no aggregates exhibited excessive ACR expansion.

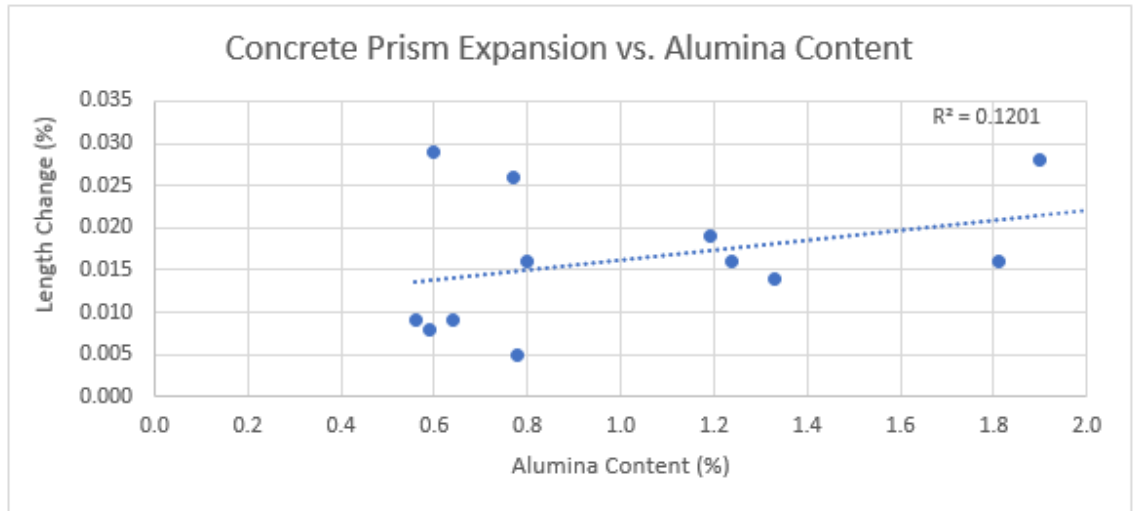
Table 4. Concrete prism test results

Sample ID	Source	Aggregate Type	Length Change (%)
C4467	Kentucky	Siliceous Limestone	0.016
C4477	Kentucky	Siliceous Limestone	0.026
C4483	Kentucky	Siliceous Limestone	0.028
C4510	Missouri	Siliceous Limestone	0.016
C4533	Kentucky	Siliceous Limestone	0.029
C4573	Missouri	Oolitic Stone	0.016
C4608	Kentucky	Limestone	0.009
C4652	Texas	Limestone	0.005
C4705	Kentucky	Siliceous Limestone	0.014
C4706	Alabama	Limestone	0.008
C4707	Alabama	Limestone	0.009
C4709	Illinois	Siliceous Limestone	0.019

Relationship Between ACR Expansion and Clay Content

The relationship between the concrete prism test results and the aggregate's clay contents (estimated based on their alumina content) was plotted in Figure 4. Based on this plot, a poor correlation was observed between the clay contents and the 12-month length change data, with a low R^2 value of 0.12. This suggests that the clay content did not play an overarching role in expansion from the selected aggregate sources.

Figure 4. Relationship between ACR expansion and clay content



Conclusions

This study aimed to evaluate whether an aggregate's clay content plays an overarching role in ACR expansion and deterioration within the limestone sources that have been approved for use in portland cement concrete in Louisiana. A total of 29 aggregate sources from DOTD's approved materials list were screened for chemical analysis to determine whether the aggregates were potentially expansive based on their calcium oxide, magnesium oxide, and alumina contents. Twelve out of 29 aggregates were found to be potentially reactive and therefore were selected for concrete prism tests per ASTM C1105 to verify their expansive potential.

The results showed that none of the aggregates tested exhibited deleterious expansion after 12 months, with only two aggregate sources near the 0.030% expansion threshold. In addition, the clay content (based on the aggregate's alumina composition) did not directly affect the selected group of aggregates' reactivity. However, given the complex nature of ACR expansion, and the inherent variability within carbonate rocks, more research is needed to conclusively determine whether an aggregate's clay content has an effect on ACR, specifically on carbonate rock sources that are compatible with portland cement concrete based on their soundness, strength potential, and durability index.

Recommendations

The results from this study indicate that DOTD's carbonate aggregate approval process for the inclusion on the approved materials list is adequate, and no modifications to the approval process are recommended at this time.

Acronyms, Abbreviations, and Symbols

Term	Description
AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
ACR	Alkali-Carbonate Reaction
ASR	Alkali-Silica Reaction
cm	centimeter(s)
FHWA	Federal Highway Administration
ft.	foot (feet)
in.	inch(es)
DOTD	Louisiana Department of Transportation and Development
LTRC	Louisiana Transportation Research Center
lb.	pound(s)
m	meter(s)
mm	millimeter(s)

References

- [1] P. Mehta and P. Monteiro, *Concrete Microstructure, Properties, and Materials*, 4th Edition, New York, NY: McGraw-Hill, Inc., 2014.
- [2] D. Hadley, "Alkali Reactivity of Carbonate Rocks— Expansion and Dedolomitization," in *40th Annual Meeting of the Highway Research Board*, Washington, D.C., 1961.
- [3] M. Tang and Y. Lu, "Rapid Method for Determining the Alkali Reactivity of Carbonate Rock," in *7th International Conference on Alkali-Aggregate Reaction in Concrete*, Ottawa, Canada, 1986.
- [4] ACI Committee 221, "ACI 221.1R-98: Report on Alkali-Aggregate Reactivity," American Concrete Institute (ACI), Farmington Hills, MI, 1998.
- [5] E. Swenson, "A Reactive Aggregate Undetected by ASTM Tests," *ASTM Bulletin*, vol. 226, pp. 48-51, 1957.
- [6] D. Stark, "Handbook for Identification of Alkali-Silica Reactivity in Highway Structures," Strategic Highway Research Program, National Research Council, Washington, D.C., 1991.
- [7] G. Wong, "Investigation of Concrete from I-20 Near Monroe, Louisiana," US Army Corps of Engineers , Vicksburg, MS, 1998.
- [8] Louisiana Department of Transportation and Development (LADOTD), *Louisiana Standard Specifications for Roads and Bridges*, Baton Rouge, LA: Louisiana Department of Transportation and Development (LADOTD), 2016.
- [9] AASHTO, AASHTO PP65-11: Standard Practice for Determining the Reactivity of Concrete Aggregates and Selecting Appropriate Measures for Preventing Deleterious Expansion in New Concrete Construction, Washington, D.C.: American Association of State and Highway Transportation Officials, 2011.

- [10] E. G. Swenson and J. E. Gillott, "Alkali Reactivity of Dolomitic Limestone Aggregate," *Magazine of Concrete Research*, vol. 19, no. 59, pp. 95-104, 1967.
- [11] J. Farny and B. Kerkhoff, "Diagnosis and Control of Alkali Aggregate Reaction in Concrete," Portland Cement Association, Skokie, IL, 1997.
- [12] M. Ozol, "Alkali-Carbonate Rock Reaction," in *Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169D*, West Conshohocken, PA, ASTM International, 2006, pp. 410-424.
- [13] M. Beyene, J. Munoz, R. Meininger and A. Shastry, "Reactive Phases in Alkali Carbonate Reaction Reference Aggregate," *ACI Materials Journal*, vol. 117, no. 6, pp. 281-292, 2020.
- [14] M. Tang and M. Deng, "Progress on the Studies of Alkali-Carbonate Reaction," in *12th International Conference on Alkali-Aggregate Reaction in Concrete*, Beijing, China, 2004.
- [15] D. Hadley, "Alkali Reactivity of Dolomitic Carbonate Rocks," *Highway Research Record*, vol. 45, pp. 1-19, 1964.
- [16] M. Ozol, "Alkali Carbonate Rock Reaction," in *ASTM STP 169C: Significance of Tests and Properties of Concrete and Concrete-Making Materials*, West Conshohocken, PA, ASTM International, 1994, pp. 372-387.
- [17] M. Tang, Z. Liu and S. Han, "Mechanism of Alkali-Carbonate Reaction," in *7th International Conference on Alkali-Aggregate Reaction in Concrete*, Ottawa, Canada, 1986.
- [18] L. Tong, "Alkali-Carbonate Rock Reaction," Thesis, Nanjing Institute of Chemical Technology, Nanjing, China, 1994.
- [19] M. Tang and M. Deng, "Progress on the Studies of Alkali-Carbonate Reaction," in *12th International Conference on Alkali-Aggregate Reaction*, Beijing, China, 2008.

- [20] A. Lopez-Buendia, V. Climent, M. Urquiola and J. Bastida, "Influence of Dolomite Stability on Alkali-Carbonate Reaction," in *13th International Alkali-Aggregate Reactions Conference*, Trondheim, Norway, 2008.
- [21] J. Gillott, "Mechanism and Kinetics of Expansion in the Alkali-Carbonate Rock Reaction," *Canadian Journal of Earth Sciences*, vol. 1, no. 2, pp. 121-145, 1964.
- [22] E. Swenson and J. Gillott, "Alkali-Carbonate Rock Reaction," *Highway Research Record*, vol. 45, pp. 21-40, 1964.
- [23] J. Gillott and E. Swenson, "Mechanism of Alkali-Carbonate Rock Reaction," *Quarterly Journal of Engineering Geology*, vol. 2, no. 1, pp. 7-23, 1969.
- [24] T. Katayama, "How to Identify Carbonate Rock Reactions in Concrete," *Materials Characterization*, vol. 53, no. 2-4, pp. 85-104, 2004.
- [25] T. Katayama, "Modern Petrography of Carbonate Aggregates in Concrete Diagnosis of So-Called Alkali-Carbonate Reaction and Alkali-Silica Reaction," in *8th CANMET/ACI International Conference on Recent Advances in Concrete Technology*, Montreal, Canada, 2006.
- [26] T. Katayama and H. Sommer, "Further Investigation of the Mechanisms of So-Called Alkali-Carbonate Reaction Based on Modern Petrographic Techniques," in *13th International Conference on Alkali-Aggregate Reaction in Concrete*, Trondheim, Norway, 2008.
- [27] T. Katayama, "The So-Called Alkali-Carbonate Reaction (ACR) – Its Mineralogical and Geochemical Details, with Special Reference to ASR," *Cement and Concrete Research*, vol. 40, no. 4, pp. 643-675, 2010.
- [28] P. Grattan-Bellew, L. Mitchell, J. Margeson and N. Deng, "Is Alkali-Carbonate Reaction just a Variant of Alkali-Silica Reaction, ACR=ASR?," *Cement and Concrete Research*, vol. 40, no. 4, pp. 556-562, 2009.
- [29] M. Beyene, A. Snyder, R. J. Lee and M. Blaszkiewicz, "Alkali Silica Reaction (ASR) as a Root Cause of Distress in a Concrete Made from Alkali Carbonate

- Reaction (ACR) Potentially Susceptible Aggregates," *Cement and Concrete Research*, vol. 51, pp. 85-95, 2013.
- [30] P. Fecteau, B. Fournier, M. Choquette and J. Duchesne, "Contribution of the Understanding of the So-Called Alkali-Carbonate Reaction," in *14th International Conference on Alkali-Carbonate Reaction*, Austin, TX, 2012.
- [31] W. Dubberke and V. Marks, "The Relationship of Ferroan Dolomite Aggregate to Rapid Concrete Deterioration," Iowa Department of Transportation, Ames, IA, 1987.
- [32] C. Rogers, P. Grattan-Bellew, R. Hooton, J. Ryell and M. Thomas, "Alkali-Aggregate Reactions in Ontario," *Canadian Journal of Civil Engineering*, vol. 27, no. 2, pp. 246-260, 2000.
- [33] J. Smeltz and J. Farver, "Dedolomitization and Alkali Reactions in Ohio-Sourced Dolostone Aggregates," Ohio Department of Transportation, Columbus, OH, 2017.
- [34] T. Prinčič, P. Štukovnik, S. Pejovnik, G. De Schutter and V. B. Bosiljkov, "Observations on Dedolomitization of Carbonate Concrete Aggregates, Implications for ACR and Expansion," *Cement and Concrete Research*, vol. 54, pp. 151-160, 2013.
- [35] H. Lee, R. D. Cody, A. M. Cody and P. G. Spry, "Observations on Brucite Formation and the Role of Brucite in Iowa Highway Concrete Deterioration," *Environmental & Engineering Geoscience*, vol. 8, no. 2, pp. 137-145, 2002.
- [36] Z. Xu, X. Lan, M. Deng and M. Tang, "A New Accelerated Method for Determining the Potential Alkali-Carbonate Reactivity," *Cement and Concrete Research*, vol. 32, pp. 851-857, 2002.
- [37] S. Stokowski and J. Sarson, "Alkali Carbonate Reactivity in Concrete," Stone Products Consultants, Westport, MA, 1997.
- [38] M. Beyene and R. Meininger, "Alkali Reactive Carbonate Rocks: Is it Alkali Silica Reaction (ASR) or Alkali Carbonate Reaction (ACR)?," in *6th International Conference on Durability of Concrete Structures*, Leeds, United Kingdom, 2018.

- [39] M. Tang, M. Deng, Z. Xu, X. Lan and S. Han, "Alkali-Aggregate Reactions in China," in *10th International Conference on AAR in Concrete*, Melbourne, Australia, 1996.
- [40] V. Jensen, "Alkali Carbonate Reaction (ACR) and RILEM AAR-1 Petrographic Method," in *12th Euroseminar on Microscopy Applied to Building Materials*, Dortmund, Germany, 2009.
- [41] M. Tang, M. Deng, X. and Lon and S. Han, "Studies on Alkali-Carbonate," *ACI Materials Journal*, vol. 91, no. 1, pp. 26-29, 1994.
- [42] K. Mather, A. Buck and W. Luke, "Alkali-Silica and Alkali-Carbonate Reactivity of Some Aggregates from South Dakota, Kansas, and Missouri," in *Symposium for Highways Research, Highway Research Board*, Washington, D. C., 1964.
- [43] A. Buck, "Investigation of a Reaction Involving Non-Dolomitic Limestone Aggregate in Concrete," US Army Corps of Engineer, 1965.
- [44] E. G. Swenson and J. E. Gillot, "Characteristics of Kingston Carbonate Rock," in *39th Annual Meeting of the Highway Research Board*, Washington, D.C., 1960.
- [45] G. Topličić-Ćurčić, V. Mitić, D. Grdić and M. Miljković, "Alkali-Aggregate Reactions in Concrete," in *3rd Advanced Ceramics and Applications Conference*, Belgrade, Serbia, 2016.
- [46] T. Katayama, "A Critical Review of Carbonate Rock Reactions – Is Their Reactivity Useful or Harmful?," in *9th International Conference on Alkali-Aggregate Reaction in Concrete*, London, England, 1992.
- [47] ASTM, C1778-20: Standard Guide for Reducing the Risk of Deleterious Alkali-Aggregate Reaction in Concrete, West Conshohocken, PA: ASTM International, 2020.
- [48] Canadian Standards Association (CSA), "CSA A23.2-26A: Determination of Potential Alkali-Carbonate Reactivity of Quarried Carbonate Rocks by Chemical Composition," Canadian Standards Association (CSA), Mississauga, Ontario, Canada, 2014.

- [49] ASTM, C1105-08a(2016): Standard Test Method for Length Change of Concrete Due to Alkali-Carbonate Rock Reaction, West Conshohocken, PA : ASTM International, 2016.
- [50] ASTM, C295/295M-19: Standard Guide for Petrographic Examination of Aggregates for Concrete, West Conshohocken, PA : ASTM International, 2019.
- [51] C. Milanesi, S. Marfil, O. Batic and P. Maiza, "The alkali-carbonate reaction and its reaction products, an experience with Argentinean dolomite rocks," *Cement and Concrete Research*, vol. 26, pp. 1579-1591, 1996.
- [52] ASTM, C856-20: Standard Practice for Petrographic Examination of Hardened Concrete, West Conshohocken, PA: ASTM International, 2020.
- [53] C. Rogers, "Evaluation for the Potential for Expansion and Cracking of Concrete Caused by the Alkali-carbonate Reaction," *Cement, Concrete and Aggregates*, vol. 8, no. 1, pp. 13-23, 1986.
- [54] ASTM, C586-19: Standard Test Method for Potential Alkali Reactivity of Carbonate Rocks as Concrete Aggregates (Rock-Cylinder Method), West Conshohocken, PA: ASTM International, 2019.
- [55] L. Dolar-Mantuani, "Expansion of Gull River carbonate rocks in sodium hydroxide," *Highway Research Record*, vol. 45, pp. 178-195, 1964.
- [56] H. H. Newlon, W. C. Sherwood and M. A. Ozol, "A Strategy for Use and Control of Potentially Reactive Carbonate Rocks," Virginia Council of Highways Investigation and Research, Charlottesville, VA, 1972.
- [57] J. Ryell, B. Chojnacki, G. Woda and Z. Kognuszy, "The Uthoff quarry alkali-carbonate rock reaction – a laboratory and field performance study," *Transportation Research Record*, vol. 525, pp. 43-53, 1974.
- [58] C. Rogers, "Alkali-Aggregate Reaction in Ontario," in *Seventh International Conference on Alkali-Aggregate Reaction in Concrete*, Ottawa, Canada, 1987.

- [59] H. Walker, "Chemical Reactions of Carbonate Aggregates in Cement Paste," in *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, West Conshohocken, PA, ASTM International, 1978.
- [60] A. Buck, "Control of Reactive Carbonate Rocks in Concrete," U.S. Army Corps of Engineers, Vicksburg, MS, 1975.
- [61] P. Smith, "Learning to live with a reactive carbonate rock," *Highway Research Record*, vol. 45, pp. 23-27, 1964.
- [62] B. Mather, "Developments in Specifications and Control," *Transportation Research Record*, vol. 525, p. 38-42, 1974.
- [63] ASTM, ASTM C233/C233M-18: Standard Test Method for Air-Entraining Admixtures for Concrete, West Conshohocken, PA : ASTM International, 2018.

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