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**ALKALI - AGGREGATE REACTION
IN NOVA SCOTIA
IV. CHARACTER OF THE REACTION**

by
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ALKALI-AGGREGATE REACTION IN NOVA SCOTIA
IV. CHARACTER OF THE REACTION

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(Communicated by G. M. Idorn)

ABSTRACT

Studies of an alkali-aggregate reaction in Nova Scotia have shown that it differs in important characteristics from the well-known alkali-silica reaction. It is discussed in relation to previously published concepts of mechanism and an explanation is proposed for the alkali-expansivity of some Appalachian rocks.

Des études d'une réaction alcaline des agrégats en Nouvelle-Ecosse - voir les articles déjà publiés - montrent qu'elle diffère sur des points importants de la réaction alcaline des silices, bien connue. Elle est examinée à la lumière de notions mécaniques déjà publiées et l'on propose une explication de l'expansion en alcalins de certaines roches appalachiennes.

Three previous papers (1, 2, 3) describe an alkali-aggregate reaction occurring in Nova Scotia that differs in important respects from the well-known alkali-silica reaction. In this fourth paper of the series a comparison is made between the two and a mechanism is suggested to account for the Nova Scotia reaction. The scanning electron microscope and an x-ray diffraction method were used to identify minerals and to follow the changes that occurred in the reaction.

The alkali-expansive rock types in Nova Scotia are mainly greywackes, argillites, phyllites, and some of the quartzites and schists. Two previously reported cases, also in the Appalachian region and both apparently involving phyllites, would not appear to be related (4, 5), as may well be a reported instance in the Canadian Shield (6). Certain phyllites in Norway and Sweden are believed responsible for alkali-expansivity of concrete (7, 8).

Present Hypotheses

The literature indicates general agreement that excessive expansion of concrete resulting from alkali-silica reaction is caused by the swelling of a gel, produced by reaction between the alkalis deriving from the cement and certain active siliceous minerals and rock types, e. g., opal, opaline cherts and rocks containing an amorphous or low crystalline phase of silica (7 to 14). Swelling pressures are believed due to the imbibition of water by the gel and this can exceed the tensile strength of mortar or concrete (15, 16).

Differences of opinion exist on some details of the mechanism: the role played by lime and the relative swelling pressure of the alkali-silica gel and the lime-alkali-silica gel; the existence of a semi-permeable membrane and its role in developing an osmotic force; and, the importance of rate of diffusion and the factors governing it.

Evidence has been presented (8, 17, 18) that swelling occurs in the outer shell of aggregate particles and that this is responsible for the ultimate breakdown of concrete.

The alkali-carbonate rock reaction is a more recent discovery. Although it shares some major characteristics of the alkali-silica reaction, and several attempts were made to put it into the same class of reaction, the differences were so marked that the same mechanism of reaction was clearly not applicable. In this case certain dolomitic limestones used as coarse aggregate have been shown to react with cement alkalis to produce excessive expansion of concrete. The expansion was early ascribed to the dolomite crystals themselves, to an osmotic mechanism, and to complex salt formation (14). More recent studies (19) offer evidence that expansion results from moisture uptake by the clay or clay-like component following a dedolomitization reaction.

Experimental Methods

A suite of 4 greywackes, 3 phyllites, 2 argillites, 4 quartzites and 1 rhyolite were selected from the many rocks tested in this study for more detailed investigation. Included were both expansive and non-expansive Nova Scotia rocks. Length change data, x-ray diffraction analyses, optical micrographs, and scanning electron micrographs were obtained. The length change characteristics of the rocks immersed in alkali were determined in cylinders by a test modified from that described in ASTM C-586 and also by means of Tuckerman extensometers. The x-ray results were recorded from rock slices and size-fractionated powders. Comparison slices of rock were analysed at increasing time intervals of treatment in water and 2M NaOH; powders were treated in $MgCl_2$, NaCl, NaOH and KOH. Fracture and saw-cut surfaces were examined in the electron microscope. The same areas of surface were observed in the untreated condition and after increasing time intervals in alkali (20).

The data obtained in this series of experiments on the Nova Scotia rocks, and the results of others, cannot be recorded here because of space limitation but are drawn upon in this paper.

Characteristics of the Nova Scotia Reaction

All alkali-aggregate reactions appear to have the common response to alkalis, moisture, and temperature, and in severe cases lead to ex-

pansion and cracking of concrete. The Nova Scotia reaction responds in like manner and, furthermore, is suppressed by the presence of a pozzolanic material as in the case of the alkali-silica reaction.

The main alkali-expansive rock types of Nova Scotia are of sedimentary origin: greywackes, argillites, and phyllites. These rocks contain little or no opal, vitreous silica or volcanic glass. Fine quartz, which is a common constituent, frequently showed signs of strain, inclusions, intergrowth and sutured boundaries. The resulting defect molecular packings on the surface probably constitute a significant proportion of the total quartz volume. Although such material may be a causative agent of expansive alkali-silica reaction, this has yet to be demonstrated through experiment. This fine quartz is the only mineral present in significant quantity, however, that can be related to the reactive component of the classical alkali-silica aggregates.

In the alkali-silica reaction a "pessimum" proportion of active mineral or rock type, in the order of about 5 per cent of the total aggregate, has been reported to give maximum expansion (21). The percentage composition at which the pessimum is found, however, is thought to be dependent upon particle size, alkali contents and type of reactive constituents. For example, dense Danish flints may show a pessimum at 100 percent under some conditions whereas it may occur at 2 percent for opaline Danish flint (G. Idorn private communication). For the Nova Scotia rock types that exhibited excessive alkali-expansivity there appeared to be a maximum expansion when the percent of expansive rock type ranged from 35 to 100 percent. The heterogeneous nature of these rocks would, however, preclude any quantitative interpretation of such results.

A temperature inversion at about 130° F, noted in mortar bar or concrete prism tests in both the alkali-silica and in the alkali-carbonate rock reactions, was not observed in the present case. "Rims" surrounding reactive aggregate particles, to some degree emphasized in the alkali-silica reaction concretes, were observed infrequently in the affected Nova Scotia concretes, and then in both affected and unaffected concretes.

This has also been observed by Idorn (8).

Excessive expansion of Nova Scotia concretes, in laboratory and field, was accompanied by considerable gel formation in some cases and practically none in others. Unaffected concretes sometimes contained little gel and others large amounts. In some field concretes that had expanded and cracked, whitish deposits in cracks and cavities were clearly the products of leaching. Analyses of random samples of these gels gave compositions that were comparable with those produced by the alkali-silica reaction.

The ASTM Quick Chemical Test, C289, was carried out on 14 selected Nova Scotia rock types: 6 greywackes, 1 rhyolite, 3 quartzites, 1 argillite, 2 phyllites and 1 schist. This method is based on reaction between alkali and active silica. By expansion tests, 8 were highly alkali-expansive, 2 moderately so, 2 slightly so, and 1 non-expansive. Another laboratory carried out the quick chemical tests on a similar set of Nova Scotia rock types. The 25 test results obtained agreed with the alkali-expansivity based on length change in 13 cases and disagreed in 12 cases, tantamount to a guess. Agreement was obtained in the case of the rhyolite and two of the quartzites and the argillite.

With the exception of rhyolite and alkali-expansive Nova Scotia rock types expand when immersed in NaOH. In the case of the rhyolite and other selected rocks known to be alkali-silica reactive, there was an actual contraction when immersed for extended periods in NaOH, e.g., chalcedony, agate, flint (see Table I). The latter behaviour would appear to be due to the loss of solid silica by dissolution in the alkali.

It was found that Nova Scotia alkali-expansive rocks showed a slight weight loss of about 2 per cent when immersed in 1.0M NaOH for 120 weeks. By comparison a chert lost 24 per cent and a flint 37 per cent in 120 weeks; an agate lost 35 per cent of its weight in 62 weeks.

In one experiment rock cylinders of alkali-expansive Nova Scotia rocks were immersed for 72 weeks in various concentrations of NaOH at 100° F. Figure 1 shows typical results for a greywacke. The expansions

TABLE I

Typical Volumetric Expansions with Time of Cylinders of Nova Scotia Rock Types Immersed in 1.0M NaOH at 73° F Compared with Volume Changes of Rock Types Previously Recognized as Potentially Alkali-Silica Reactive.

Rock Type	Vol. exp., per cent, at weeks shown					
	4	12	48	72	96	120
N.S. Quartzitic greywacke	.002	.005	.047	.096	.159	.222
N.S. Calcareous argillite	.011	.016	.051	.060	.067	.074
N.S. Phyllite	.036	.037	.104	.159	.239	.286
N.S. Micaceous quartzite	.004	.000	.014	.064	.138	.200
Chalcedony (Riversdale)	.013	.003	-.043]	disintegrating		
Agate (Brazil)	.036	.043	-.036]			
Flint (England)	.007	.002	.035	.022	-.013	-.237

Figure 1

Time-mean volumetric expansion relationships for greywacke AP24/S4 showing continued expansion in distilled water at 100° F after initial curing in various NaOH solutions at 100° F

increase with increasing alkali content. After 72 weeks the cylinders were removed from alkali and placed in water for 56 weeks. During this time there was a slight drop in rate of expansion but it did continue. Either the alkali had effected an irreversible change which allowed continuing uptake of water with consequent expansion, or the alkali cannot be readily dislodged from the rock element with which it is reacting. Hence regeneration of the alkali by the calcium ion, considered by some to be necessary in the alkali-silica reaction (10), appears not to be required in the Nova Scotia reaction.

The above differences in behaviour of the Nova Scotia alkali-expansive rocks from those variously claimed of the classical alkali-silica reaction strongly suggested that another mechanism must be operative, at least in part. It was particularly important to account for continuing expansion in highly water-leachable conditions, such as obtained in hydraulic structures in Nova Scotia where expansion was shown, by actual measurement on the structures, to be continuing after 30 years (22). This slow but continuing expansion over many years has since been noted in at least one case in the Canadian shield (private communication from the Hydro-Electric Power Commission of Ontario).

It is certain that relatively low average annual temperatures play a role in the rate of reaction, but laboratory studies demonstrate that expansion is very slow and generally does not level off even after three years under accelerated conditions.

Microscopy and X-Ray Analysis

Petrographic descriptions of the four main rock types - greywackes, quartzites, phyllites and argillites have been given in an earlier paper (2). The chief minerals present are framework silicates (quartz and feldspars) and sheet silicates ($10\text{\AA}$ - mica, chlorite, vermiculite or vermiculite-type mixed layer mineral and a smectite that is probably montmorillonite or a closely related mineral). Accessories are various iron minerals, graphite and tourmaline. The proportioning of these constituents varies with the nature of the rock and some of the minerals are not always present.

Rock wafers were immersed in 2M NaOH at 100°F and x-ray diffraction patterns recorded before and after treatment with alkali. Diffractograms of many of the rocks showed a shift in peak position from about 10Å to about 12.6Å (Figure 2, a to d).

X-RAY: $\text{CuK}\alpha$, 45KV, 25mA, $1^\circ 2\theta / \text{MIN}$.

Figure 2

X-Ray diffractograms of phyllite and greywacke wafers before and after soaking in 2M NaOH at 100°F

Examination was made on the scanning electron microscope of the same area of rock surface before and after treatment with alkali (20). In some samples exfoliation of layer-structure silicates was observed (Figure 3, c and d), followed at later ages by complete removal of these minerals. New rhombic or cubic crystals within the laminae were also sometimes present. The layer-structure silicate which showed exfoliation was correlated by size to the brown pleochroic crystals observed optically and believed to be mainly a vermiculite-type mineral. Similarly this mineral was believed responsible for the shift from about $10\text{\AA}$ to $12.6\text{\AA}$ in peak position observed on x-ray diffractograms of alkali-treated rocks. In some cases branching structures were visible on the alkali-etched surface, the material either having underlain the phyllosilicates or formed from them due to the alkaline attack (Figure 3, e and f). The four main rock types also contained layer-structure silicates which were apparently not affected by alkali while the rock specimens were immersed (Figure 3, a and b).

Discussion and Conclusions

Much of the data on the expansion of rocks, concretes and mortars described in the first three papers in this series (1, 2, 3) were derived by conventional test methods accelerated variously by elevated temperatures and increased alkali contents. Early work in acceleration of slow reactions was reported by Carlson (23) who also tested a greywacke and a phyllite. It is emphasized again that in the Nova Scotia studies conclusions were based on such results only if corresponding evidence had been obtained by non-accelerated experiments and field evidence.

A survey of the literature indicates that most of the observations used in developing hypotheses for mechanism of alkali-silica reaction were founded on studies of alkali reaction with opal-bearing rocks (e.g., (16)). The theories proposed appear to account satisfactorily for expansion of this and related minerals or rock types. In the study of the Nova Scotia alkali-aggregate reaction, and in some previously reported studies involving phyllites, argillites and related non-carbonate sedimentary rocks used as concrete aggregate, there is strong evidence that the mechanisms previously proposed for the alkali-silica reaction are not entirely applicable.

Figure 3

Scanning electron micrographs of greywacke (no. 9), phyllite (no. 3), and argillite (no. 4) before and after treatment with alkali

Many of the Nova Scotia rocks used as concrete aggregates contain a smectite related to montmorillonite and porphyroblasts of a brown pleochroic mineral that looks like biotite under the optical microscope but is identified by x-ray analysis to be vermiculite or a closely related mixed-layer mineral. Scanning electron micrographs show that some of the phyllosilicates exfoliate when treated with caustic soda solution.

The shift in peak position on x-ray diffractograms from $10\text{\AA}$ to $12.6\text{\AA}$ that resulted from treatment of the rock with NaOH demonstrates expansion of the mineral. The spacing conforms fairly closely to that reported by Brown (24) for air-dry Na-vermiculite at room temperature. In water, however, larger spacings of about $14.8\text{\AA}$ are quoted. The spacing of the mineral in the Nova Scotia rock wafers failed to increase to a value greater than $12.6\text{\AA}$ even after prolonged soaking in 2M NaOH at 100°F , and even though the wafers were saturated and x-rayed under high relative humidity conditions. A strong peak at $14 - 15\text{\AA}$ was, however, registered on diffractograms recorded from oriented powders of many of the ground, dispersed and sized rocks which had been treated with solutions of NaOH. Possibly the acid, H_2O_2 and the NaOH used during dispersion and sizing of the powdered rock affected the mineral so that greater expansion was possible. The minerals in oriented powder mounts are also free from restraints that may normally restrict the expansion of minerals in rocks.

Collapsed smectites also have a basal spacing of about $10\text{\AA}$ that under certain conditions increases to about $18\text{\AA}$ or more. It is possible that the shift in peak position on the x-ray diffractograms resulted from expansion of the smectite as well as the vermiculite, but the spacings recorded are closer to those normally attributed to vermiculite than montmorillonite. The size of the exfoliated mineral on the scanning electron micrographs also correlates with the vermiculite observed by optical microscopy.

Complexes of hydrated aluminum and iron have been reported to occur in the interlayer space of minerals that normally show variable basal spacing (25), (26). This interlayer precipitate sometimes interferes with the expansion and contraction characteristics of the mineral. Its properties

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are said to be intergradient between those of vermiculite, chlorite and montmorillonite. It seems possible that similar interlayer precipitates of poorly organized silica may exist.

Interlayer precipitates have been extracted by a variety of reagents that have included a mixture of KOH plus KCl (27) and boiling NaOH (25). If such precipitates exist between the sheets of vermiculite and montmorillonite in the Nova Scotia rocks, it is probable that they would be removed to a greater or lesser extent by an alkaline solution as obtains in a normal concrete mix. Once this precipitate was removed the mineral would be capable of expansion through water adsorption and the force would be transmitted to the rock. Hence the volume of the rock should increase. Extraction by cement alkali of such interlayer complexes may also lead to precipitation elsewhere in the concrete of gel-like secondary products. Some gel may also be derived by dissolution of fine lattice defect quartz that may or may not contribute to rock expansion as postulated in earlier hypotheses. In any case the presence of such gels would account for the assumption of "classical" alkali-silica reaction commonly made in routine evaluations of deteriorated concretes containing this type of reactive aggregate.

Diffraction patterns recorded from rock wafers soaked in hot water for periods of a year or more showed no shift in peak position. This may indicate that the interlayer precipitates are insoluble in hot water and are extracted only by the much more aggressive 2M NaOH.

The energy released through chemisorption of water molecules on an active surface, particularly if newly formed, has been shown in the clay and cement literature to be quite high and analyses and equations have been proposed for the pressures exerted by adsorbed water in swelling materials (28, pp. 107 - 113).

In the case of the alkali-expansive rocks in the present study the layered-structures were observed, on a micro-scale, in an apparently "collapsed" condition. Soaking in alkali produced an increasing exfoliation with time. Since this was actually observed in a rock where restraints are high, it follows that the pressures exerted by such exfoliation must have

been great enough to produce expansion of the rock. The relatively small separation of layers and the type of bonding normal to an active surface would maintain the pressure in the system. This direct evidence, though not quantitative is of minimum magnitude and therefore adequate in the present argument.

The above concepts explain the long-term expansion of field concretes in water-leachable environments and the continued expansion of rock cylinders in water after prior treatment in alkali (Figure 1). The rate of dissolution of these "cementing precipitates" may in large part determine the slow rates of reaction observed in the field and laboratory. Once alkali has freed-up the layers, water imbibition and expansion should continue for a time period that will depend on such factors as moisture availability, permeability, internal restraint, and the quantity and distribution of potentially expansive mineral.

It is concluded that there are extractable interlayer precipitates between the basal planes of the vermiculite-like mineral in alkali-expansive Nova Scotia rocks and that once this precipitate is removed by NaOH the mineral expands and causes an increase in the volume of the rock. There are small amounts of smectite present which also may contribute to expansion.

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