Salt Weathering of Limestone Aggregate and Concrete Without Freeze-Thaw

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Kansas aggregates are frequently alkali reactive or subject to D-cracking when used in concrete. The use of deicing salt, usually halite (NaCl) in Kansas, makes those problems worse. Salt allows the concrete to become wet and to stay wet longer, increasing the time for reactions to occur. Clays in limestone aggregates have been altered by deicing salt solutions remaining in the aggregates. Degraded illite changed to sodium montmorillonite. Quartz has been altered by electric currents induced in the concrete. Some quartz took on an optical property, undulatory extinction, that is frequently associated with potential alkali reactivity. Salt (NaCl) scaling of concrete blocks and slabs without freeze-thaw has been observed. Monitoring salt water movement through the walls of concrete cups has provided insight on how and where salt water moves in concrete. The salt water movement and deposition of salt has caused considerable scaling of both the cement paste and the limestone aggregates of the concrete. The salt "gnaws away" or corrodes the limestone aggregate and cement paste, attacking the most accessible and most susceptible parts first. Particles as large as 0.6 in. have been observed in aggregate cups. Most of the scaled flakes are oatmeal size and no larger than 0.2 in. in length. A silane "sealer" did not prevent salt water from moving through the limestone aggregates in the concrete cups. All three cups treated with silane cracked on the first salt treatment cycle (five days filled with salt water, emptied, soaked in plain water, then nine days of air drying). Untreated cups did not crack even after 12 cycles. No freeze-thaw was involved.

Unlike many states, Kansas does not have an abundance of good native mineral aggregates for concrete and therefore must make do with what is available. Sand, sand-gravel, and crushed limestone are the three most commonly available Kansas aggregates. The state uses the crushed chat (chert) tailings from the lead and zinc mining operations in southeast Kansas. Gravel, crushed sandstone, and silicified chalk have been used. Expanded shale, a man-made, lightweight, glassy aggregate that is highly porous, resembling scoria or pumice, has also been used.

Gravels, sand-gravels, and sand may contain reactive quartz, glassy volcanics, opaline, chaledonic, cherty, or other particles that are alkali reactive. The silicified chalk is quite variable and frequently contains reactive opaline and low chertostalite-tridymite material. Silica-cemented sandstone may also be reactive due to the same substances. The calcite-cemented sandstone, sometimes locally called quartzite, is usually not reactive, but in places the cement is dolomitic, which may cause problems through the alkali-carbonate dedolomitization process. A few beds of fine-grained dolomite have been used as crushed stone, and they too suffer that reaction. The expanded shale aggregates are sometimes prone to an alkali-aggregate reaction that has caused growth of bridge decks. Many lightweight decks had several inches of length (as much as 14 in.) sawed off to accommodate the expansion reaction and growth. Most Kansas limestone units contain beds that produce D-cracking under freeze-thaw conditions. Some gravel and sand-gravel sources have contributed to D-cracking.

Because of all their mineral aggregate problems, Kansas expended considerable effort during the 1930s, 1940s, and 1950s sorting out sources of acceptable and unacceptable material for concrete. The strategy became one of avoiding reactive or tested sources of potentially reactive aggregates, or requiring replacement of 30 to 35 percent of the coarse aggregate fraction with crushed limestone — a process called sweetening (1).

About 30 years ago Kansas began using deicing salt regularly. It was soon recognized that deicing salt was aiding and abetting concrete deterioration. Alkali-aggregate reaction and D-cracking came on quicker than before salt was used. By then, however, its use to provide bare pavements had become entrenched in the traveling public's mind. Salt continued to be used as a "safety agent" no matter what researchers, materials engineers, or environmentalists said about the detrimental effect of salt on bridges, pavements, or the roadside and subsurface environment. Reevaluation of materials became necessary. There was controversy over whether the problems were primarily physical, chemical, both, or neither. Some maintained that there was no problem. Yet many concrete roads and bridges were rapidly decaying. There was no lack of deteriorated concrete to study. The financial burden to repair or replace prematurely damaged roads, bridges, roadside plantings, and water wells across the country is strongly felt by taxpayers and public servants alike.

CLINICAL OBSERVATIONS

Several observations have been made relating to the various ways salt seems to affect Kansas mineral aggregates and the concrete containing them. This report does not address salt-induced corrosion of reinforcing steel. Salt is hygroscopic and absorbs moisture from humid air. This enhances the alkali-aggregate reaction, which generally does not proceed if the
internal relative humidity of the concrete is below about 80 percent (1). When enough salt is present, the concrete can become saturated at relative humidities below 80 percent (2).

Even in the reasonably dry Kansas environment the near-ground atmosphere usually contains considerable moisture during the early morning hours, before the hot sun begins surface evaporation. During the day the exposed surfaces dry as the early morning dew evaporates. The internal moisture in the concrete is slow to leave. Salt increases the dwell time for moisture in the concrete. This gives added time for the alkali-aggregate reactions to proceed as the concrete warms in the sun. Hence salt increases the chances for concrete to become wet and stay wet internally. If the concrete contains reactive aggregates, the chances for reaction are increased. Salt also provides almost daily cycles of surface wetting and drying, as well as warming and cooling of exposed concrete.

Alkali carbonate deicing salts can add alkali to the concrete (3) to further aggravate the reaction problem.

Alkali-carbonate reactions were observed to occur in a four-year-old Kansas concrete pavement. The pavement had been built using a crushed stone coarse aggregate composed of a fine-grained dolomite. The pavement was near Kansas rock salt mines and was deiced regularly during the winter with the local rock salt. Stains and surface dampness were frequently observed near sawed transverse joints, especially at their intersection with longitudinal joints. Cores obtained in those locations revealed little sign of internal cracking or deterioration. However, the copper nitrate staining technique recommended by Dolar-Mantuani (4, 5) revealed that the fine-grained porous dolomite was converting to calcite in the border areas of the dolomite aggregate. After a few more years the joints required repair because they had begun to deteriorate.

Salt was helping maintain a high internal humidity. During the warm months an alkali-carbonate dedolomitization reaction was occurring. This was augmented by D-cracking of the same aggregates during the winter. The sand-gravel used with the dolomite was not observed to be reactive. This fact, and the fact that the concrete contained only 36 percent crushed dolomite, allowed the road to be maintained for most of its 20-year design life by repairing the joints as deterioration progressed. Some blowups occurred on hot days after rain.

A report of a Kansas example of map cracking progressing into D-cracking in a limestone-sweetened reactive aggregate concrete pavement was published by TRB several years ago (5). Deicing salt exacerbated the problem with alkali-aggregate reaction and pavement blowups occurring during the warm months and D-cracking during the winter. At the end of 11 years only 3 percent of the joints were still good. Map cracking, D-cracking, and pavement blowups are all enhanced by the hygroscopic action of salt, which absorbs moisture from the air and keeps the concrete more saturated.

Hudec and Rigby (6) found that immersing carbonate aggregates in a 3 percent sodium chloride solution doubled their later water absorption at 30°C and 98 percent relative humidity, compared to unsalted specimens. Under immersion conditions the degree of saturation increased an average of about 13 percent for the salted rocks. Many of the individual rock samples showed considerably more or less than the average increase. Hudec and Rigby found little effect at 45 percent relative humidity. They did not report any other levels of relative humidity tests.

Under conditions of wetting and drying, warming and cooling, or freezing and thawing, the water inside the concrete and its porous aggregates shifts from small spaces to larger spaces, from the inside to the surface, and from pores to cracks. When the conditions reverse, the movement of moisture also reverses direction. Thus the moisture in the concrete moves about with changes in temperature, humidity, wind velocity, vapor pressure, rainfall, sunshine, electrical current, and so forth.

As the water moves, it dissolves or deposits substances depending on internal conditions. On a microscopic level, conditions inside salted, moist concrete seem to be continually changing with changing ambient conditions. When moisture moves between paste and limestone aggregates, the border zone of each is somewhat altered. The aggregate may develop negative or positive rims (3, 7, 8) that are often made up of multiple layers. Air voids, cracks, and other voids in concrete also frequently contain multilayered coatings inside the voids, attesting to many alternating cycles of water movement and deposition due to changing conditions. Any petrographer who has examined much in-service concrete has observed these deposits.

Efflorescence or fibrous deposits on the external surfaces of concrete often give clues to changing internal conditions that may involve the mineral aggregates. Such was the case of soft, fluffy, fibrous, white deposits collected from the bottom of an old bridge deck. Hot mix asphalt covered the full bridge deck, which was being evaluated for probable replacement. X-ray, microscope, and chemical studies of the soft white deposits revealed that they were aphthitalite (K, Na)2SO4 with thernardite Na2SO4.

The deck had been routinely deiced with rock salt that contained from 2 to 7 percent sulfate, present mostly as anhydrite (9). Thus the sodium sulfate was not surprising. However, there was little potassium in the deicing salts tested, and the authors therefore believed the potassium must have been derived from concrete constituents. They found that the limestone aggregates in the concrete above the fibrous deposits were often badly deteriorated. Bukovatz et al. described the concrete as “sea of deterioration” with occasional “islands” of sound concrete (10, Figure 4). X-ray studies of the clay fraction of the cracked and broken limestone aggregates revealed that much of the degraded illite normally present had altered to sodium montmorillonite. Sodium from the NaCl had exchanged with potassium in the degraded illite. It was concluded that most of the potassium in the aphthitalite was from that source. There were indications that alkali-feldspars in those “seas of deterioration” were also weathering under the highly saline freeze-thaw conditions created by deicing salts penetrating the badly cracked hot mix asphalt overlay to the old non-air-entrained concrete below. The concrete seldom had a chance to dry out thoroughly because the hot mix retarded moisture evaporation more than its penetration.

Kansas Department of Transportation (KsDOT) researchers have studied the effect of salt on indoor and outdoor exposures of concrete blocks and slabs for many years (9, 11-15). Most of that work involved freeze-thaw cycling, so the effect of salt alone could not be separated from that of freeze-thaw. In some instances, however, observations were recorded that indicated salt caused scaling before any freezing cycles had occurred (13).

In electro-migration studies of salt in concrete (14, 15) it was noted that the salt ions moved through the cement paste,
not through carbonate aggregates. Other studies (11) noted that the sodium in salted concrete was sometimes highest in the paste and sometimes highest in the limestone aggregate. An added "clinical observation" from the electro-migration studies of salt movement (14,15) was not included in the reports but was mentioned in an in-house memo. It was observed in petrographic studies of the electro-treated salty concrete that much of the quartz had taken on an optical characteristic often associated with alkali-reactive quartz, namely undulatory extinction. If, through electrical treatment, the quartz had become reactive, it could imply a long-term durability loss for electro-treated concrete or perhaps even concrete struck by lightning. A discussion of reactive quartz and undulatory extinction may be found in a recent work by Dolar-Mantualii (3).

In other unreported KsDOT work, the authors pursued scaling studies of concrete blocks subject to surface salting under outdoor exposure. The blocks contained no reinforcing steel and were made such that one-half of each block contained air-entrained concrete and the other half contained non-air-entrained concrete of the same mix. Companion blocks were made for exposure without salt applications. The cured blocks were 75 days old when they were moved outdoors in late spring of 1983 after all danger of frost was past (all blocks were supported on wooden frames). When the blocks were three months old, 300 ml of a 3 percent NaCl (laboratory grade) solution was applied three times a week to the test blocks. The same amount of salt-free water was applied to the companion controls at the same time.

During the first week it was necessary to spread the liquids on the block surfaces because they tended to bead up and not flow over the concrete. After the first week (three treatments) the salt solution quickly spread over the entire surface of the blocks when applied. The plain water continued to bead up and required spreading effort by rubber spatula to cover the block surface. It was noted that the salt-treated blocks took far more time to surface dry than did the blocks treated with water. As treatments progressed, the surface drying time differential increased.

After nearly five months of outdoor exposure, 49 salt applications had been made but no freezing had occurred. All of the salt-treated blocks had very shallow surface flaking, whereas the control blocks treated only with water did not. The air-entrained portion of the salt-treated blocks had slightly less surface scaling than did the non-air-entrained portions. The 49 salt applications would roughly equate to 2½ years of Kansas road salting, according to McCollom (16). There would not be 2½ years of road salting without freeze-thaw in Kansas, however. At an average of more than 68 freeze-thaw cycles per winter in Kansas, 2½ years of salting would represent about 170 cycles of freeze-thaw. Another difference in these tests is the lack of sulfates and other impurities normally present in Kansas rock salt. The salting of the blocks continued through the winter months and the scaling increased considerably but mostly in the non-air-entrained half of the salt-treated blocks.

SALTY CONCRETE CUPS

In 1986 the authors began a study of salt movement in concrete by using cored concrete cups (17-19). They were already studying salt variations in adjacent concrete samples (20). Salt crystal growths in concrete (21,22) and simpler or more rapid means of determining salt content in concrete (23,24) were topics of other salt studies in KsDOT labs.

For making concrete cups (Figure 1), two concentric cores are taken in the concrete. The smaller, shallower inner core is drilled first and broken out before the larger outer core is drilled to a greater depth—often through the full thickness of concrete (17). The cored cups are rinsed and allowed to air dry before being used for salt movement studies. In order to obtain cups with walls ½ in. thick, the authors used a core bit with a 2½ in. outside diameter for the inner core or "well" of the cup. The larger core bit is 3 in. in inside diameter. Other sizes and combinations may be used. For variable thickness walls the authors used a bit of 4 in. inside diameter for the larger core. By offsetting the larger core to one side of the inner core, they obtained cup walls that range from ½ in. to about 1½ in. thick in the same cup. All cups have a bottom from ½ in. to several inches thick.

A 15 percent salt solution made from standard Kansas deicing salt was used. This concentration was chosen because it was used in the NCHRP 244 studies (25). It is also the concentration used many decades ago for NaCl durability studies of limestones (26). It was reported that Indiana limestone suffered seven times as much deterioration from the salt durability tests as from freeze-thaw action. In the present studies, the 15 percent salt solution was used to fill the concrete cups to within about ½ in. of the top.

Moisture may show up on the outside of the cup in a matter of minutes (2 to 20 minutes), but it is usually several hours to a day before salt crystals are deposited on the outside of the concrete cup (17-19). Figure 2 shows the outside of a cup nearly two weeks after salt water was added. There are NaCl crystal deposits at the perimeter of many of the limestone aggregates. Aside from cracks, the coarse limestone aggregate border is the zone of most rapid salt movement. Some of the limestone particles become coated with salt, indicating more rapid flow through certain aggregates than through others. Earlier work with field concrete revealed a sodium chloride buildup in some of the limestone coarse aggregates in a bridge deck (11). In studies of D-cracking in Kansas concrete pavements, the authors often observed from core samples that many aggregates were sound and that only a portion of the limestone coarse aggregates were deteriorating. Those were the most susceptible aggregates. The authors now wonder if our salt cup tests are identifying some of those most susceptible individual aggregate particles.

FIGURE 1 A group of concrete cups made by taking two concentric cores such that the cup walls are about ½ in. thick and the bottom is at least ½ in. thick. 

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The photograph in Figure 2 also shows general surface dampness on the outside of the cup. This is moisture adsorbed from the air. On humid days the salt drew the moisture from the air and the cup surface remained damp as long as the humidity was high. This did not occur on untreated cups. On days when it was raining outdoors, the moisture buildup on the outside of the cups was often great enough to dissolve some of the salt deposits. The water ran down the outside of the cups and collected in the glass plates containing the cups. Thus, periods of very high humidity sometimes obliterated the original depositional pattern of the salt on the outside of the cups.

At the top of the cup in Figure 2 is a mass of salt around the inside perimeter. This is from salt water wicking up the inside walls, evaporating, and leaving the halite crystals behind. Figure 3 shows this buildup in an early stage. Rounded masses of halite crystals appear first. Needle crystals of salt then often grow from those masses. More massive crystals begin to grow on the needles. As evaporation continues, the entire inside perimeter at the top of the cup becomes coated with the salt deposit, as seen in Figure 2. The needles are eventually completely overgrown and no longer show as salt deposition continues.

When salt water came through the walls of the cup, halite crystals also grew on the outside of the cup. Small oatmeal-sized flakes of paste and aggregates scaled from the cup's outside surface. Figure 4 shows a cup before salt treatment. Note especially the two limestone aggregates near the top of the cup to the left of the letter H. Figure 5 is a close-up of the larger of those two aggregates after salt treatment. Notice the surface cracking and thin flakes pushing out of that aggregate. Salt crystals are growing below the surface and causing surface flaking or scaling. Figure 6 shows the same aggregate after the surface had flaked off and the salt deposits were dissolved by gentle immersion of the emptied cup in plain water.

The authors cycled the first two cups through a dozen salt water treatments. Each cycle consisted of five days with 15 percent salt water in the cups, followed by emptying, gentle soaking in plain water to remove the salt deposits, and air drying for nine days. More oatmeal-sized flakes were dislodged by the salt crystal growths during each cycle. Figure 7 is a close-up of some clumps of those flakes, the largest of which is 0.2 in. long. Figure 8 shows the largest individual scaled flake collected, which is 0.6 in. in length. The flake is...
FIGURE 5 The larger of the two aggregates noted in Figure 4. Salt has moved through the aggregate, depositing near its top surface. The salt is also pushing thin flakes from the surface of the aggregate.

FIGURE 6 The aggregate particle in Figure 5 after the salt has scaled away part of the surface. A distinct rim shows in the second aggregate slightly above and to the right. The limestone in the rim has been separated from the aggregate particle by salt weathering.

FIGURE 7 Typical scaled particles of paste and limestone aggregates. These range up to 0.2 in. in longest dimension.

FIGURE 8 A bean-shaped flake of aggregate 0.6 in. long. This is the largest flake that remained in one piece after being scaled off by the salt. The aggregate this came from may also be seen in Figures 2, 10, 11, and 12.

about $\frac{1}{16}$-in. thick and includes the surface of a limestone aggregate particle.

Figure 9 shows the surface of a cup after 12 salt cycles. The paste phase has scaled or flaked away and the coarse aggregate stands out in relief. The coarse aggregates shown in this photograph did not scale as much as the paste, but other aggregates do scale as seen in Figures 5, 6, and 8. All this was from salt scaling without any freeze-thaw. The cups and salt water were kept at ambient laboratory temperature of 68 to 74°F.

Many of the aggregates that scaled did so at their perimeter, adjacent to the zone of salt water movement at the aggregate borders. Figure 10 is the same cup as in Figure 2, but the cup has been slightly rotated in a clockwise direction for the photograph of Figure 10. This picture was taken after the salt water was emptied from the cup, the salt deposits dissolved by immersion in fresh water, and the cup allowed to air dry. Note the two aggregates near the top of the cup, just to the left of the letter A. (Three letters were placed about 120
FIGURE 9 A concrete cup that has gone through 12 cycles. Here the aggregates were more durable; the cement paste has weathered away and the limestone aggregates stand out in relief.

Figure 11 shows the same area of the cup in Figure 10 after several more cycles of salt water treatment, immersion wash, and air drying. Note the perimeter of the two aggregates at the top of the cup to the left of the A. Both aggregates have a rim that has separated from the aggregate. This type of rim eventually flakes away, leaving a depressed border zone in the aggregate. At the center of Figure 11 are two elongated limestone aggregate particles with their long axis nearly vertical in the cup wall. The smaller of the two, a bean-shaped aggregate, is the flake shown in Figure 8. Figure 12 is the same general shot as Figure 11 after the flake in Figure 8 had scaled off; the pencil points to the spot where the flake came from. Other aggregates in both Figures 11 and 12 also have rims separating from the aggregate proper. A similarly scaled rim can be seen in the top right corner of Figures 5 and 6. Many of the same aggregates shown in Figures 10, 11, and 12 may also be seen in the right half of Figure 2. The location of the salt deposits are visible in Figure 2. After 12 cycles, about 95 percent of the limestone aggregates in this cup had been scaled to some extent.

Figure 13 shows a limestone coarse aggregate where the entire rim has flaked away, leaving the aggregate depressed compared to the cement paste. This is the opposite of the condition shown in Figure 9, where the paste has flaked away and the aggregates stand out in relief. The salt attacks the most susceptible component of the concrete—sometimes the paste, sometimes the aggregates, sometimes both. No two concretes are alike, but salt is persistent in finding a weakness to attack.
Sometimes the salt water found preexisting cracks in the limestone aggregate particles. Figure 14 shows such a particle with salt deposits along cracks in the aggregate. Under freeze-thaw conditions this aggregate would probably have deteriorated from water freezing in the cracks.

One cup that went through several cycles of treatment began developing a rust deposit on the face of a limestone aggregate particle during the sixth cycle. This corrosion continued and ran down the inside face of the cup. The authors do not yet know what mineral in the aggregate is corroding, but they presume it is an iron oxide or an iron sulfide. Corrosion of minerals in the aggregates is another effect of salt weathering.

Three cups were treated internally with an alkyl-alkoxy silane type “sealer” such as used in the NCHRP 244 studies (25). This sealer did not prevent salt water movement through the limestone aggregates (19). One surprising result, however, was the cracking of all three silane-treated cups during the first cycle of salt water treatment. None of the nonsealed cups cracked even after as many as 12 cycles. Figure 15 shows one of the silane-treated cups with a pattern of cracks that goes through some limestone aggregates but alongside others. These were not the aggregates that the salt water passed through. Halite crystals at the top of the cup were deposited from evaporating salt water that wicked up the inside walls of the cup.

SUMMARY AND CONCLUSIONS

Many “clinical observations” made by KsDOT research geologists, as well as laboratory and field investigations, have provided information on how deicing salt (NaCl) weathers concrete and the aggregate in it. Salt exacerbates the alkali-aggregate reaction, D-cracking problem, and pavement blowup problem by keeping the concrete interior wet for longer periods of time. When the concrete and limestone aggregates remain wet, the dwell time of salt solution in the aggregates increases. This allows more time for other reactions to occur.

Degraded illite altered to sodium montmorillonite in limestone aggregate in Kansas bridge deck concrete. Sodium from
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