Evaluation of Remolded Field Samples of Lime-Cement-Fly Ash-Aggregate Mixtures

ERNEST J. BARENBERG, University of Illinois

In late season construction using lime-cement-fly ash-aggregate (LCFAA) mixtures, the reaction process may be greatly retarded by cool temperatures. There is some concern that as free water percolates through the pavements under these conditions there will be a tendency for the lime and cement to migrate and/or be leached from the mixture. If such is the case, and if a significant quantity of water percolates through the mixture, there may not be sufficient lime and cement available for proper reaction when the temperature rises the following spring.

Construction of the runways and taxiways at the Newark Airport provided an excellent opportunity to collect the necessary materials for a study to determine if lime and cement does migrate or is leached from the materials during adverse climatic conditions. Placement of the LCFAA material was started in late summer 1968 and continued through late November. During the latter portion of the construction season, a significant amount of rain fell on newly constructed pavements. Also, the mean daily temperature in the project area during the latter part of the 1968 construction season was in the low to mid 40's, which is sufficiently low so that little or no reaction would be expected to occur in these materials.

Construction procedures used in the Newark Airport were sufficiently controlled so that reasonable uniformity could be expected in the LCFAA mixtures. Layers of compacted LCFAA mixtures were placed in layers up to 30 in. thick and provided an excellent opportunity to study the migration of lime and cement throughout the depth of the pavements as the water percolated through the material. Also, the LCFAA materials used had sufficient permeability so that the water could percolate freely through the material thus providing ideal conditions for leaching to occur.

A laboratory investigation was designed to determine the extent of leaching or migration of the lime and cement under the field conditions described and the effect of the leaching and curing conditions on the subsequent curing of the LCFAA materials.

MATERIALS

Field samples of the LCFAA materials were taken from the pavement locations given in Table 1. Samples of the coarse aggregate, sand, and fly ash were obtained from stockpiles of materials used in the construction of these facilities. Characteristics of the coarse aggregate, sand, and fly ash are given in Table 2. Samples of lime were obtained from the suppliers and included both high calcium and monohydrated dolomitic limes. Portland cement used in the study was obtained from local supplies.

Field samples were taken from the airport pavements in 2-in. increments throughout the depth of the pavements. Thicknesses of the materials in place varied from 6 to 30 in. (Table 1).

When collecting the field samples, the material was placed in double plastic bags and carefully sealed to prevent moisture loss during shipment. The samples were shipped to the laboratory by commercial truck. The samples were shipped during early January and were frozen when received. They were stored in the frozen condition until preparation for testing to prevent further reaction of the constituents.

---

Moisture content determinations were made on all samples as obtained from the field. A 600-gram sample was taken from each field sample for titration and pH tests to determine the lime and cement contents. The remainder of the material was set aside for strength and durability testing.

Mix formulas for the materials used in the pavement construction are given in Table 3. Optimum moisture contents and maximum dry densities for these mixtures are also given in Table 3. All mix proportions are given on a dry-weight basis.

TEST PROGRAM

The test program consisted of (a) titration and pH tests on all field samples (60); (b) strength and durability tests on selected samples of remolded material (30); and (c) a laboratory study to evaluate leaching under controlled conditions. Miscellaneous tests as necessary to develop the standards for comparison were also conducted.

TEST PROCEDURES

Titration Test

A titration procedure was used to determine the lime and cement content of the field samples. Details of the procedure are presented elsewhere (1). Essentially the procedure consists of dissolving the calcium (and magnesium) from the mixture in a solution of ammonium chloride and then determining the calcium concentration in the

### Table 1

<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Pavement Station</th>
<th>Centerline Reference</th>
<th>Mix Designation</th>
<th>Depth (in.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4L-22R 50+00</td>
<td>70 ft W</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 24</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>24 to 30</td>
</tr>
<tr>
<td>2</td>
<td>Taxiway B 10+00</td>
<td>On centerline</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 24</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>24 to 30</td>
</tr>
<tr>
<td>3</td>
<td>4L-22R 49+25</td>
<td>70 ft E</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 22</td>
</tr>
<tr>
<td>4</td>
<td>Taxiway A 10+00</td>
<td>10 ft E</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 24</td>
</tr>
<tr>
<td>5</td>
<td>Taxiway A 10+00</td>
<td>10 ft W</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 24</td>
</tr>
<tr>
<td>6</td>
<td>Taxiway A 58+00</td>
<td>5 ft W</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 24</td>
</tr>
<tr>
<td>7</td>
<td>Taxiway A 10+00</td>
<td>10 ft E</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 24</td>
</tr>
<tr>
<td>8</td>
<td>Taxiway B 56+50</td>
<td>25 ft W</td>
<td>A</td>
<td>0 to 8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>B</td>
<td>8 to 16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>16 to 24</td>
</tr>
<tr>
<td>9</td>
<td>Taxiway A 37+00</td>
<td>10 ft W</td>
<td>A</td>
<td>0 to 8</td>
</tr>
</tbody>
</table>

Note: Loss on ignition of fly ash was 10 percent.
solution by titrating with a disodium salt of EDTA (ethylenediaminetetraacetic acid). The calcium concentration in solution is determined by comparing the EDTA necessary to titrate the solution from the field samples with a standard curve prepared from mixtures with known concentrations of lime and cement. Separate calibration curves are required for each mix formula and each lime type. Calibration curves for the materials used in this study are available in Appendix A.

**pH Determination**

Fifty grams of material from the field samples were charged into a plastic vial and approximately 100 ml of distilled water was added. After vigorous shaking for approximately 1 minute, the sample was allowed to settle for approximately 2 minutes and the pH of the fluid then read with a direct reading, electrode-type pH meter.

**Strength Test**

Unconfined compression tests were conducted on 2- by 4-in. cylindrical specimens made from remolded samples of material taken from the pavements. The specimens were cast in a solid steel mold and compacted in 3 approximately equal layers with a falling mass impact hammer with a 2-in. diameter face. The compacted specimens were pushed from the mold, cured in a sealed plastic bag at the prescribed temperature for the prescribed period of time, soaked, capped with hydrocal, and tested at a strain rate of 0.05 in. per minute.

Material for the cylindrical specimens was prepared by mixing for approximately 3 minutes in a Lancaster counter current mixer with a muller attachment. Water was added during mixing as required to bring the material to the desired (optimum) moisture content. The muller attachment effectively pulverized any clunks of material present in the field samples of the LCFAA material. Field samples with moisture contents above optimum were dried back to just below optimum prior to mixing. Laboratory prepared mixtures, which were used for comparison purposes, were blended and dry-mixed for 1 minute before adding water and 2 minutes after water was added.

**Durability Test**

The durability characteristics of the field material were evaluated by evaluating the potential of remolded field samples to develop and retain compressive strength after a short curing period and repeated freeze-thaw cycles.

Six 2- by 4-in. cylindrical specimens were made using the procedure outlined for the compressive strength test. After compaction these specimens were scaled in a plastic bag and cured for 2 days at 120 F. Three specimens were then removed and tested in compression, and the remaining 3 specimens were bedded in moist sand and placed in a freezer for 16 hours. After 16 hours in the freezer the specimens were removed and allowed to thaw for 8 hours at approximately 70 F. During the thawing period the sand in which the specimens were embedded was kept very moist, approaching saturation. After the prescribed number of freeze-thaw cycles, the embedded specimens were covered with plastic to prevent moisture loss, stored for 7 days at 120 F, then soaked for 4 hours, capped with hydrocal, and tested in compression.

**Leaching Test**

A test program was undertaken to evaluate leaching of the lime and cement from the LCFAA mixtures under laboratory conditions. Four lucite tubes, approximately 2 ft
long and 4 in. in diameter, were filled to a depth of approximately 18 in. with a labora-
tory prepared mixture (Mix B). The mixture was brought to optimum moisture content, and the proper quantity of material was charged into a lucite tube and vibrated to the
desired density. After compaction, specimens were covered and allowed to cure for
approximately 48 hours at a temperature of approximately 70 °F. After 48 hours of cur-
ing 2 samples were treated with a prime coat (MC-O applied at a rate of 0.25 gal/sq yd), and all 4 specimens were placed in a controlled temperature cabinet and held at a tem-
perature of between 35 and 40 °F.

Water in appropriate quantities to simulate the rain that fell on the pavements was
poured on each specimen and allowed to percolate through the LCFAA material. The
water was placed on the specimens in quantitative increments to simulate the amount of
rain that fell during specific periods on the pavements. For example, on November 10,
1968, approximately 1 in. of rain fell on the pavements between 1 a.m. and 4 p.m. To
simulate this condition sufficient water was placed on the laboratory samples at 7 a.m.
to give the equivalent of ½ in. of rainfall; at 11 a.m. water equivalent to an additional ½
in. of rain was added, and the same amount was added again at approximately 4 p.m.
It was observed that all water from previous applications had completely seeped away
prior to all subsequent applications. All water that percolated through the mixtures
was caught, retained, and tested for calcium content using the titration test.

When the amount of water equivalent to the rainfall for a 10-day period after place-
ment had percolated through the samples, the material was removed from the tubes and
subjected to the same test program as the field samples.

PRESENTATION AND DISCUSSION OF FINDINGS

Data from tests on field samples are given in detail in Tables B-1 through B-6 in
Appendix B. Some of these data are reproduced in different format in the body of this
report to facilitate the discussion.

Leaching of Lime and Cement

It is expected that if lime and cement were leached from the LCFAA mixtures it
would produce several significant changes in the material. If leaching is fairly com-
plete it can be expected that the pH of the material will drop to below the 12.0 to 12.3
range (2). It is also expected that with significant leaching there would be a marked
reduction in the lime plus cement content as determined by the titration test. It also
seems reasonable to expect that, if sufficient calcium is leached from materials in the
upper layers to produce a calcium-saturated water percolating through the material,
some of this calcium will be deposited in the lower layers thus producing an increase
in the indicated lime plus cement content of the material from the lower depths.

A study of the pH data from the field samples shows that the pH of only 3 samples fell
below 12.0, and only one of these had a pH below 11.0. Generally, free lime still
is present in the material and available to react under favorable conditions.

Results from the titration tests on the field samples are shown in Figure 1. Two
curves are shown: The curve for the lower lime plus cement content is based on a 1-
minute stirring time in the NH₄Cl, and the curve for the higher lime plus cement con-
tent is based on a 5-minute stirring time. These 2 stirring times were used in lieu of
the standard 2-minute stirring time in an effort to distinguish between the lime and ce-
ment that was readily dissolved in the NH₄Cl, and that which required a longer dissolu-
tion time. As seen in Figure 1, the curves for the 1- and 5-minute stirring times are
similar in shape and, for practical purposes, are parallel.

Two features are noted in the data shown in Figure 1. First, the lime plus cement
contents determined by the titration tests are generally lower than the lime plus cement

---

1See footnote 1.
content specified for these materials, and, second, the lime plus cement content determined with a 5-minute stirring period is consistently higher than that determined with the 1-minute stirring period. These data suggest that not all lime and cement are dissolved by the NH₄Cl, at least not during the relatively short mixing times used in this study. There is also a question of how much influence the curing time and temperature have on these results.

To develop further information on the effect of curing and stirring times on the indicated lime plus cement content, a series of specimens of mix B were made and cured for varying times in sealed bags at 120 F. These specimens were then pulverized in the same manner as the field samples and the lime plus cement contents determined using the titration test procedure. Results from this study are shown in Figure 2. In general, it is seen that as the curing time increases the lime plus cement content indicated by the titration procedure decreases. The reduction in the indicated lime plus cement content is especially rapid during the first 2 days of curing. Results shown in Figure 2 also indicate that as the curing time increases longer stirring times are needed to remove the comparable quantities of lime and cement from the mixture. The indicated lime plus cement content after 4 to 7 days of cure in the laboratory at 120 F is about the same as that obtained from the field samples.

Results from the leaching study on the material in the lucite tubes in the laboratory also indicate a relatively small amount of calcium is leached from the samples. The lime plus cement contents determined by the titration tests on the laboratory specimens are given in Table 4. The average lime plus cement contents of 1.95 percent with 1-minute stirring and 2.22 percent with 5-minute stirring are not significantly different from lime plus cement contents obtained from the field samples using identical test procedures.
Titration tests on the water that percolated through the LCFAA materials in the tubes indicated that less than 0.1 percent (of the approximately 4.0 percent added to the mixture) of lime plus cement had been dissolved in the water as it percolated through the mixture.

**Strength and Durability Test Results**

Durability as used in this presentation refers to the ability of the material to regain or retain compressive strength under repeated cycles of freezing and thawing and subsequent curing. Thus, strength and durability of these materials are closely related, and the test results are discussed together.

Figure 3 shows the age-strength relationship of LCFAA mixtures prepared in the laboratory. The 2 lower lines represent the data from the original mix design on these materials (3), and the upper line is from a mixture made and cured in the laboratory using the mix B formula. Numbers in the parentheses on the 2 lower lines denote the ratio of the strength of the material after it had cured for 1 day at 120 F and was then remolded and recompacted to that of the undisturbed material.

The effect of time between placement and sampling and subsequent remolding on the strength development of LCFAA field samples is shown in Figure 4. These data indicate that as the period between placement and sampling increased the strength developed in

---

**TABLE 4**

<table>
<thead>
<tr>
<th>Tube</th>
<th>Percent Lime Plus Cement Content After Mixing in NH₄Cl</th>
<th>1 Minute</th>
<th>5 Minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.9</td>
<td>1.0</td>
<td>2.0</td>
</tr>
<tr>
<td>2</td>
<td>2.1</td>
<td>2.1</td>
<td>2.5</td>
</tr>
<tr>
<td>3</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>4</td>
<td>1.8</td>
<td>1.8</td>
<td>2.4</td>
</tr>
<tr>
<td>Average</td>
<td>1.96</td>
<td>2.00</td>
<td>2.225</td>
</tr>
</tbody>
</table>
Figure 3. Relationship between curing time and compressive strength and between remolded and undisturbed samples.

Figure 4. Effect of time between mixing and placing and remolding on the compressive strength of the LCFAA materials in the field.
the remolded material decreased. This is as expected, as the more lime and cement utilized in the reaction process before remolding the less there is available to react after remolding and recompaction. This should be kept in mind in evaluating the subsequent data on the strength development of remolded and recompacted materials from the field samples.

Figure 5 shows the effects of alternate freeze-thaw cycles and accelerated curing on the strength development of the laboratory prepared LCPAA mixture. When the material has had a relatively short curing time, alternate cycles of freezing and thawing may cause a significant reduction in the compressive strength. This strength is quickly recovered, however, when curing conditions are favorable as is shown in Figure 5 for the second 2-day curing period. After the second curing period, the material gained strength during the freeze-thaw cycles and had a further substantial gain in strength during the 2-day curing period following the second series of freeze-thaw cycles.

Figure 6 shows the strength gain and loss pattern for the undisturbed laboratory mix of LCPAA material plus a probable curve of the disturbed material developed from tests using materials from the field samples. The strength of the remolded material from the field samples after 7 freeze-thaw cycles was not determined as the upper \( \frac{1}{8} \) to 1 in. of the specimens appeared soft so that effective capping was not possible. After an additional 7 days curing at 120° F, the softened materials rehardened sufficiently to permit the specimens to be capped and tested in compression.

The strength of the remolded field samples of LCPAA material after 2 days curing at 120° F was of the order of one-half the strength of freshly mixed material for the same curing conditions. There is no direct comparison that can be made of relative strengths of the undisturbed and remolded materials after the freezing and thawing, but examination of the trends of the data indicates that the remolded materials did develop a substantial proportion of their potential strength as indicated by the results from tests on
Figure 6. Comparison of durability of disturbed and undisturbed samples.

Figure 7. Correlation between lime plus cement content and compressive strength of field samples.
the undisturbed material. Considering the effect of time between compaction and re-molding on the field samples, these results suggest that the materials in place should develop adequate strength and durability under favorable curing conditions during the spring and summer months.

Figure 7 shows the relationship between the lime plus cement content indicated by the titration test and the compressive strength developed in the remolded field samples. The natural scatter in the test results plus the limited number of test results available make it impossible to establish a precise correlation. There appears, however, to be a strong trend toward higher strengths with higher indicated lime plus cement contents.

CONCLUDING REMARKS

Findings from this program indicate that the lime and cement in the LCFAA mixtures used in the Newark Airport pavements are stable and are not subject to significant leaching or migration over short periods of time. More specifically, the findings indicate that those LCFAA materials placed late in the construction season will retain nearly all of the lime and cement that were added during mixing and that this lime and cement will react with other components of the mixture under favorable conditions even after being dormant for a considerable period.

Findings from this study indicate that the more extensive the curing is before freezing and thawing the less damage will be done to the materials by cyclic freezing and thawing. Also, materials damaged by freezing and thawing will recover a significant portion of the lost strength under favorable curing conditions provided sufficient lime and cement are retained in the mixture to support continuing reactions. It was observed in this study that, when the LCFAA materials are subjected to alternate freeze-thaw cycles under the conditions used in this study, the most severe damage to the materials occurs near the top of the specimens for a depth of from 1 to 2 in.

Findings from this study seem to indicate that LCFAA materials can be placed later in the construction season than previously thought, provided proper precautions are taken. For example, it may be necessary that for late season construction the lime or lime and cement contents be increased to offset the effects of any initial set and damage caused by freezing and thawing to the ultimate strength developed by the LCFAA mixture. Also, provision should be made to remove any loosened or damaged materials from the surface prior to placing subsequent layers during spring and summer.

ACKNOWLEDGMENTS

This study was sponsored by the Limestone Products Company, Inc., and the Warner Company, suppliers of the lime for the Newark Airport construction. The Port of New York Authority agreed to supply the field samples needed for this study. The author is grateful to these organizations for their cooperation and support for this study.

REFERENCES