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**EFFECT OF FIELD VARIABLES AND TEST PROCEDURES
ON SILANE WEATHERPROOFING PERFORMANCE**

*FINAL REPORT
FHWA/OK 93(02)*

by

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and

*THOMAS D. BUSH, JR.
Principal Investigator*

JUNE 1993

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*OKLAHOMA DEPARTMENT OF TRANSPORTATION
RESEARCH AND DEVELOPMENT DIVISION
in cooperation with the
FEDERAL HIGHWAY ADMINISTRATION*

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*FEARS STRUCTURAL ENGINEERING LABORATORY
SCHOOL OF CIVIL ENGINEERING AND ENVIRONMENTAL SCIENCE
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| ft | feet | 0.3048 | metres | m |
| yd | yards | 0.914 | metres | m |
| mi | miles | 1.61 | kilometres | km |

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|-----------------|---------------|--------|---------------------|-----------------|
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| in ² | square inches | 645.2 | millimetres squared | mm ² |
| ft ² | square feet | 0.0929 | metres squared | m ² |
| yd ² | square yards | 0.836 | metres squared | m ² |
| mi ² | square miles | 2.59 | kilometres squared | km ² |
| ac | acres | 0.395 | hectares | ha |

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| MASS (weight) | | | | |
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| lb | pounds | 0.454 | kilograms | kg |
| T | short tons (2000 lb) | 0.907 | megagrams | Mg |

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| VOLUME | | | | |
| fl oz | fluid ounces | 29.57 | millilitres | mL |
| gal | gallons | 3.785 | litres | L |
| ft ³ | cubic feet | 0.0328 | metres cubed | m ³ |
| yd ³ | cubic yards | 0.0765 | metres cubed | m ³ |

NOTE: Volumes greater than 1000 L shall be shown in m³.

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| TEMPERATURE (exact) | | | | |
| °F | Fahrenheit temperature | 5/9 (after subtracting 32) | Celsius temperature | °C |

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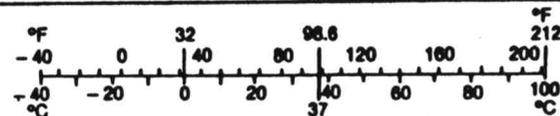
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| m ² | metres squared | 10.764 | square feet | ft ² |
| km ² | kilometres squared | 0.39 | square miles | mi ² |
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| kg | kilograms | 2.205 | pounds | lb |
| Mg | megagrams (1 000 kg) | 1.103 | short tons | T |

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| mL | millilitres | 0.034 | fluid ounces | fl oz |
| L | litres | 0.264 | gallons | gal |
| m ³ | metres cubed | 35.315 | cubic feet | ft ³ |
| m ³ | metres cubed | 1.308 | cubic yards | yd ³ |

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| °C | Celsius temperature | 9/5 (then add 32) | Fahrenheit temperature | °F |
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The contents of this report reflect the views of the authors who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views of the Oklahoma Department of Transportation or the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

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EFFECT OF FIELD VARIABLES AND TEST PROCEDURES ON SILANE WEATHERPROOFING EVALUATION

CHAPTER 1 INTRODUCTION

1.1 GENERAL

Durability of concrete bridge decks is largely dependent on exposure conditions and the concrete's ability to resist the deteriorating effects of its environment, especially water and chloride ingress (Carter 1991). In cold climates where the concrete surface is exposed to cycles of wetting and freeze-thaw, scaling may occur, particularly for concrete that has less than 5% entrained air. This situation is commonly encountered in older bridges built prior to the introduction of air entrainment specifications in standards. Chloride intrusion may also occur where de-icing salt is applied to the bridge deck. Chloride ion intrusion initiates and accelerates the corrosion of reinforcement, and the internal expansion caused by the corrosion product often causes spalling (Durability of Concrete Bridge Decks 1970).

Repairing concrete deterioration problems is usually expensive and in many cases the repair process may not be permanently effective in preventing recurrence of distress. In the past decade, a growing interest has developed in preventative measures to reduce the cost of maintenance and increase the service life of concrete bridges (Carter 1989). One focus has been the use of surface treatments to inhibit the ingress of moisture and chlorides. Several products are available; however, silicon based penetrating water repellent treatments, specifically silane and siloxane, have gained popularity because of their desirable performance characteristics. These include reduction of water and chloride ion intrusion, penetration into the concrete, moisture vapor transmissibility, and unaffected skid resistance (McGettigan 1992). The penetration of silane is important because it provides protection against wear and the deteriorating effects of UV radiation. The ability to allow moisture vapor transmission is important in preventing moisture build up in the concrete, thus reducing potential for the corrosion reaction.

Despite the growing need for and popularity of weatherproofing concrete structures, the application of silane technology has been hindered by the lack of consensus concerning standardized testing procedures. This situation is costly to the various DOT's, and is further complicated by uncertainties and concerns related to the effects of various commonly encountered field conditions

often not considered in current testing procedures. The effects of these variables on the performance of the weatherproofing material, and the improvement achieved by treating the concrete, are considerations for approval of a particular product and for determining the feasibility of the treatment process.

Many current testing procedures utilize a standard laboratory reference concrete with a specified water/cement (w/c) ratio of approximately 0.50. However, structural concrete used for bridge decks usually has a somewhat lower w/c ratio, often around 0.45. Other mixes, such as high density overlay concrete, have even lower w/c ratios. Concrete in the field may also have a variety of surface finishes, and may be subjected to a range of specified curing and cleaning procedures prior to treatment. These differences between field and laboratory conditions are usually not reflected in standard laboratory tests, and the projected performance and expected benefits based on laboratory testing may vary greatly from the field. In order to better predict field performance from laboratory tests, knowledge is needed concerning the effects of these variables on performance parameters obtained from various testing procedures.

1.2 OBJECTIVES

The objectives of this research are :

- 1) To study the effects of concrete mix design, surface finish, and curing/cleaning procedures prior to treatment on the performance of a particular silane water repellent material.
- 2) To compare parameters obtained from different testing procedures used to predict performance of silane.
- 3) To gather information to aid in selecting suitable laboratory testing procedures that could be used for laboratory screening of silane penetrating water repellent material.

1.3 SCOPE

This research is limited to studying the performance of a single "reference" silane as influenced by the variables mentioned, namely, concrete mix design, surface finish, and curing/cleaning procedures. Only laboratory tests were performed and the testing was limited to studying the following performance parameters: 1) depth of penetration of silane, 2) water absorption, 3) chloride ion intrusion, and 4) moisture vapor permeability.

Three concrete mix types were examined: class A (w/c=0.49, similar to standard laboratory "reference" concrete), class AA (w/c=0.44, similar to structural concrete), and high

density overlay concrete ($w/c=0.33$). Concrete surface conditions examined included broom and tine finishes (created by texturing fresh concrete). Curing conditions examined included laboratory (self,wet) curing, use of linseed oil emulsion curing compound, and use of a pigmented membrane curing compound. Cleaning procedures to remove curing compounds prior to treatment included dry shot-blasting and high-pressure water washing. Basic testing procedures examined included a series of tests specified by the Oklahoma Department of Transportation (ODOT), NCHRP 244 Series II cube tests (Pfieffer and Scali 1981), and a variation of the NCHRP 244 procedure. All research utilized plain concrete specimens which were not subject to any deformations, loads, or stresses.

CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

2.1 NEED FOR PROTECTING CONCRETE STRUCTURES

In a nation-wide survey (Durability of Concrete Bridge Decks 1970), three major types of bridge deck durability problems were identified:

- 1) Cracking, which is caused by shrinkage and/or temperature changes.
- 2) Spalling, which is the most expensive to repair, is either caused by the pounding action of wheel loads or by expansion of corroded reinforcements.
- 3) Scaling, due to freeze-thaw cycles.

These durability problems are often costly to repair, thus it is important to reduce the ingress of water and chloride laden water into concrete bridge decks.

Generally, it is feasible to weatherproof existing and new concrete structures when the surface is exposed to water, freezing, and salt water (Carter 1991). Studies made by Carter at the Alberta Transportation Utility indicate that unprotected bridges exposed to deicing chemicals have half the life expectancy of bridges exposed to water only. Weatherproofing improves the durability and life expectancy of the structure by reducing the ingress of water and chloride laden water into the concrete, thus reducing repair costs.

2.2 SILANE AS A SEALER

2.2.1 Available Weatherproofing Materials

Several types of materials were tested in the study presented in the NCHRP 244 report (Pfieffer and Scali 1981). These materials can be divided into two major categories:

- 1) Coating materials form a hydrophobic surface membrane that is often physically bonded to the surface of the substrate. Materials such as epoxy and polyurethane fall in this category.
- 2) Penetrants penetrate the substrate and form a hydrophobic layer. Sometimes referred to as sealers, these materials include silane and siloxane. Silane and siloxane basically differ

in their molecular structure and size. Usually a siloxane molecule consists of 2 to 12 silane molecules grouped together (Carter 1989).

Linseed oil, one of the widely used materials at the time of the NCHRP 244 report, may be categorized as a coating but it forms no well defined membrane.

In the NCHRP 244 study, several types of weatherproofing materials were tested. The results indicated that some coating materials such as epoxy provide excellent hydrophobicity but may reduce skid resistance. Epoxy has a high coefficient of thermal expansion which may inhibit successful and durable protection, and is susceptible to abrasion damage due to lack of penetration. Application of linseed oil, a widely used material, provided much less water repellency than other tested weatherproofing materials, and no measurable penetration. Siloxane provides most of the properties of silane; however, due to its larger molecular size (25-75 angstroms, as compared to 10-15 angstroms for silane) its penetration is less than that of silane (McGettigan 1992). Penetration of siloxanes is generally more sensitive to the moisture content of the substrate (Carter 1989), and chloride screening is often inferior to that of silane (McGettigan 1992).

2.2.2 Mechanism of Water Repellency

Silane is a polymer that has two important components, each with a different role in the process of weatherproofing. The components are attached to the silicon atom; the organic hydrocarbon "organofunctional" provides hydrophobicity, and the alkoxy group "silicon functional" is responsible for bonding to and penetrating into the concrete substrate (McGettigan 1992; Smith 1986). The water repellency is provided by chemically bonding the hydrocarbon molecules to the substrate which reduces the surface tension of the substrate to less than that of water (McGettigan 1992). A schematic of the molecular structure of silane is shown in Fig. 2.1.

The depth to which silane penetrates is dependent on the molecular size of silane particles, porosity of concrete, moisture and silica content, and pH of the concrete (McGettigan 1992). This penetration depth is not only important for wearing surfaces but is also important in protecting the silane from degradation due to UV radiation.

When silane is applied to the concrete it is usually dissolved in a carrier to ensure equal distribution over the area where treatment is intended. Capillary suction forces drive the solution into the concrete through the micro pores to what is sometimes called the "visible depth of penetration". The action of the capillary forces (McGettigan 1990) is the main factor that provides a high absorption rate of the silane into the concrete, which normally exceeds the rate of evaporation.

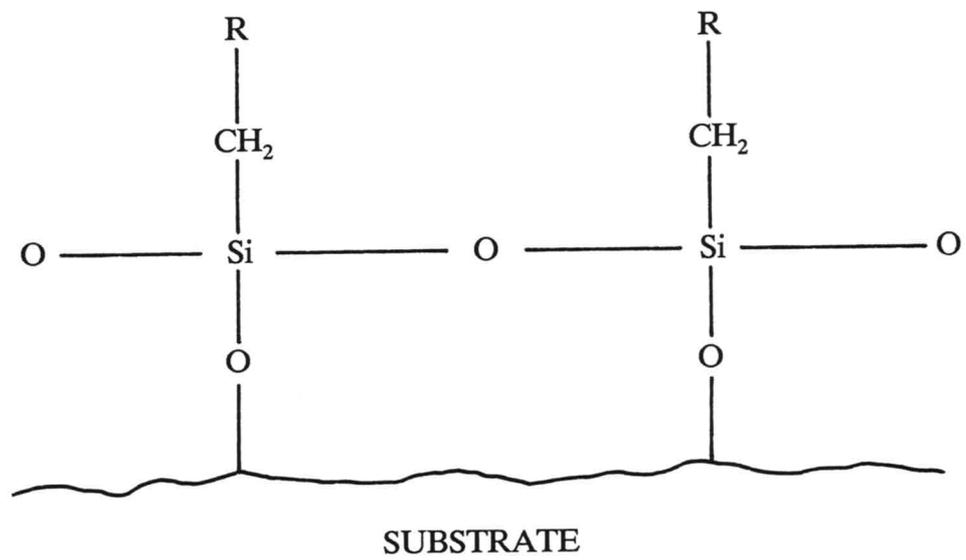


Fig. 2.1 Molecular Structure of Silane (McGettigan 1992)

As the silane is applied to the concrete surface, the silane molecules, which have hydrolyzable groups, start to react with each other and with the substrate, and hydrolysis and condensation occur (McGettigan 1992; Smith 1986). During hydrolysis, silanol molecules form in the presence of moisture, and during condensation the unstable silanol molecules bond chemically with the hydroxyl groups of the silicate molecules in the concrete. This chemical bonding is very strong and stable, unlike other weatherproofing coating materials which rely on physical bonding. The bond between silane and concrete is a Si-O bond, and that between silane and the water and chloride repellent hydrocarbon molecule is a Si-C bond. These two bond types are highly durable and stable. It is important to note that high alkalinity of the pore solution in the concrete (pH 13-13.5) can result in breaking the bond between the silane molecules and the substrate; however, this reaction is avoided by using silane with branched iso-octyl or iso-butyl alkyl groups (McGettigan 1992).

2.3 EXPERIMENTAL EVALUATION OF SILANE

2.3.1 Testing Procedures

Most testing procedures attempt to evaluate the performance of silane with respect to four basic parameters. These parameters are depth of penetration, water absorption, chloride intrusion, and moisture vapor permeability. Other tests measure parameters such as electrical potential of current produced by the corrosion process, or surface resistivity. In the past decade several researchers and organizations have developed or adopted testing procedures. Some of these are presented in the following review.

2.3.1.1 AASHTO Testing Procedures. Currently there are two testing methods adopted by AASHTO that can be used to evaluate the performance of silane. The salt ponding test, AASHTO T259-80 (AASHTO 1986b), utilizes concrete slabs exposed to 3% NaCl solution for 90 days. After the exposure period, three holes are drilled in each specimen, and powder samples are obtained at two depths from each hole. The samples are then tested in accordance with AASHTO T260-84 (AASHTO 1986c) for chloride ion concentration.

The other testing procedure is the rapid chloride permeability test, AASHTO T 277-83 (AASHTO 1986a). The resistivity of a concrete core subjected to a current passed through the specimen for 6 hours is measured. The test uses a vacuum saturation step in which the core specimen is subjected to a vacuum of 1 atmosphere while soaking in water. This point may limit the test's usefulness in evaluating penetrating sealants such as silane, since the hydrophobic behavior is largely chemical and water pressure resistance is limited.

2.3.1.2 NCHRP 244 Report. The objective of this project was to determine the effectiveness of different weatherproofing materials for concrete (Pfieffer and Scali 1981). Four testing series were performed.

In Series I, 21 weatherproofing materials were tested to determine their effectiveness in reducing chloride and water intrusion into concrete. Concrete with a w/c ratio of approximately 0.53 was used. Cubes were water cured for one week, and then lightly sandblasted, weighed, and allowed to air dry for 21 days in a standard controlled climate room. At the end of the drying period the cubes were treated and stored for 14 days prior to immersion for 21 days in a 15% NaCl solution. Another set of cubes was pretreated with linseed oil prior to treating with the tested materials. The gain in cube weight was determined at 3 day intervals during the soaking period. At the end of the soaking period the cubes were allowed to air dry in the controlled climate room and the weight loss was determined every 3 days, then at 22, 23, and 24 days of air drying. Two types of untreated control specimens were used; one type remained in air and the other underwent the same procedures as the treated specimens. At the end of the drying period each cube was split in half. One-half of each cube was crushed and the total chloride ion content was determined. Cubes treated with silane exhibited significant reduction in weight gain, and the performance of silane seemed to suffer when used with linseed oil pretreatment. The performance of silane in all cases was better than the performance of siloxane.

Series II tests were designed to determine the effect of concrete moisture content at the time of treatment upon water absorption, vapor transmission, and chloride intrusion, for five selected materials. The procedures were essentially the same as Series I tests except that cubes were cured in plastic bags and allowed to air dry for 1, 5, and 21 days, prior to treatment. Cubes underwent 21 days of air drying following the immersion period. Concrete with a 0.501 w/c ratio was used. The results indicated that the performance of silane treated cubes was not significantly affected by the length of the air drying period, for the range of drying periods studied.

Series III tests utilized the same procedures of Series II tests, with the exception of allowing all specimens to air dry for five days prior to treatment, and the application rates of the tested materials were varied to evaluate the influence of this factor on performance. It was concluded that the coverage rate did not have a significant effect on the performance of silane. The coverage rates used were the manufacturer recommended rate, and one half and double that rate.

Series IV tests utilized two accelerated weathering methods to simulate northern climate and southern climate exposure conditions. These tests used cracked reinforced concrete slabs and plain concrete slabs. The southern exposure test utilized alternate exposure to a 15% NaCl solution followed by UV light and infrared heat. The northern climate exposure test included additional

exposure elements such as fresh water rinse, acid, and freeze thaw conditions, but with reduced exposure to salt water as compared to the southern exposure test. Over the 24 week period of this test, visual observations and half cell potential measurements were made. At the end of the test, chloride intrusion was evaluated by taking drilled samples. The performance of silane in reducing chloride intrusion was observed to be 97% for southern exposure conditions and 76% for northern exposure conditions. Test results indicated the southern exposure test to be generally more severe than the northern exposure test, possibly due to the increased exposure to salt water.

The southern exposure test method was adapted and utilized (Zoob, LeClaire, and Pfeifer 1985) in a testing program specifically geared to test the performance of silane. The tests utilized concrete slabs reinforced at the top and bottom surfaces, with clear cover of 1" (25.4 mm), 2", and 3". Three types of concrete were used with w/c ratios of 0.5, 0.4, and 0.32. Also, in this testing program 48 weeks of cyclic exposure to salt water and drying were included. Half-cell potential and corrosion current were monitored. Chloride ion content was determined by taking samples horizontally drilled in the specimen sides at the level of the reinforcement when a surge was observed in the half cell potential, and after 44 weeks of testing. It is interesting to note that the chloride ion concentration was found to be higher for concrete with a w/c ratio of 0.4 than the other two concrete types for untreated concrete, and the same trend was observed for specimens treated with one type of silane. A significant reduction in chloride intrusion was observed in the specimens treated with silane. Excellent water vapor transmission was observed and this factor enhanced the corrosion protection by increasing the concrete resistance by 2 to 3 times.

2.3.1.3 Oklahoma Department of Transportation tests. The Oklahoma Department of Transportation (ODOT) has adopted three testing methods plus the salt ponding test (AASHTO T259-80) which were used in the present study. The procedures will be discussed in detail in the following chapter. These tests are:

- 1) Depth of penetration, OHD L-40 (ODOT undated, a). This test measures the depth of penetration of silane by wetting a treated broken block and observing the depth of the hydrophobic layer.
- 2) Moisture vapor permeability, OHD L-35 (ODOT undated, b). This test measures the moisture lost upon oven drying of treated specimens.
- 3) Absorption test, ASTM C 642-81 (ASTM 1987b). This test is used to evaluate the water absorption of treated specimens after immersion in fresh water for 48 hr and 50 days.
- 4) Salt Ponding Test, AASHTO T 259-80.

2.3.1.4 Alberta Test Method. The Alberta test method (Carter 1989) evolved from the NCHRP 244 series II cube test described previously. Moisture absorption is the primary measure

of performance, and absorption of sealed cubes is compared to that of unsealed cubes. The absorption comparison is done both before and after the cubes have been sand blasted to simulate wear. Vapor permeability is also measured during a drying period after the cubes have been soaked in water.

2.3.1.5 Surface Electrical Resistance Method. This recently developed test method (ASTM 1991) offers a procedure to qualitatively measure the relative effectiveness of the sealer. Two strips of conductive paint are applied to the concrete surface and the changing electrical resistance across the strips is measured. This test is one of few tests applicable in the field. Although this test is useful, it provides only limited quantitative evaluation of performance of silane.

2.3.1.6 Stand Pipe Test (Whiting, Ost, and Nagi 1991). The absorption rate of a concrete surface can be estimated by using an apparatus that consists of a small diameter tube which has an enlarged end. By attaching the larger diameter end of the tube to the concrete surface and noting the rate by which the water in the tube falls, the relative absorption can be estimated to evaluate the effectiveness of the sealer. This test method can be used in the field for vertical surfaces as well as horizontal surfaces.

2.3.1.7 Testing Methods Adopted by Highway Agencies. The use of penetrating sealers by highway agencies and testing methods adopted were surveyed in a nation-wide study (Whiting 1990). The report showed a wide variety of testing methods and varying degrees of satisfaction with the performance of the sealers, further evidence for the need for standardized and comprehensive procedures. The major tests cited included NCHRP 244 Series II, AASHTO T259 (salt ponding), ASTM C642, ASTM C672 (ASTM 1987a), AASHTO T277 (rapid chloride penetration), depth of penetration, vapor permeability, and other tests for absorption, freeze-thaw, and skid resistance.

2.3.2 Laboratory Studies

The effectiveness of sealers already in service and the effectiveness of resealing were evaluated in a study (Marusin 1989) which utilized cores taken from existing concrete structures. The cores were sliced vertically to evaluate existing chloride ion content at various depth intervals, and the depth of penetration of the sealer was evaluated. The sealer penetrated hydrophobic layer was removed from the cores which were then retreated and ponded with 15% NaCl solution. The weight gain was monitored and the chloride ion content at various depth intervals was determined at the end of the test. As expected, the chloride ion concentration decreased with depth, and the effectiveness of the sealer appeared to be affected by the quality of the concrete.

Silane has the ability of resisting chloride ion ingress to the concrete substrate (Pfieffer and Scali 1981). Yet, the concrete itself resists chlorides differently than it does water, such that the

advancing water front lies ahead of the advancing chloride front, even though the chloride ions were originally dissolved in the water (McCarter, Ezirim, and Emerson 1992). Apparently the pores in the concrete matrix provide some screening mechanism which allows the water to advance, yet at the same time resists to an extent the advancement of chloride ions. This action presumably overlaps with the action of the silane penetrated layer, which works in a similar manner. The influence of this mechanism in the concrete on the chloride penetration results obtained from silane treated samples is not clear. At low water pressure, absorption is the process responsible for water intrusion into the concrete surface, and is inversely proportional to the degree of saturation at the time of exposure. This means that concrete samples conditioned at elevated temperature in the laboratory will yield higher absorption results than under actual field conditions.

A total of 57 different weatherproofing materials were applied to specimens after immersion in a 15% NaCl solution and allowing three weeks of air drying (Aitken and Litvan 1989). Eight types of concrete were used with w/c ratios of 0.43 and 0.55, using air and water curing, with and without entrained air. The effectiveness of the weatherproofing materials was found to be dependent on the quality of concrete. In general, performance of the weatherproofing materials was most effective for water cured concrete specimens with the lower w/c ratio. The researchers also suggested that properties of the concrete must be considered when selecting a sealer for a particular application.

Studies have been conducted by the Alberta Transportation Utility (Carter 1989) for the purposes of screening weatherproofing products. Penetration has been found to be primarily influenced by substrate moisture content, molecular size of the sealer, and type of solvent used for the sealer. Performance of alcohol based products was found to be less sensitive to substrate moisture. Moisture content at time of treatment was also found to influence absorption, particularly after sandblasting to simulate surface wear. Higher moisture contents at treatment typically resulted in decreased improvement with respect to absorption. In certain cases, improvement due to treatment was actually increased after sandblasting. Apparently there is an optimum abrasion depth at which higher condensation has occurred and water repellency is greater than at the surface. This effect has been seen in studies using solvent-free (neat) silane (McGettigan 1992). It appears that absorption reduces after initial abrasion and increases after greater abrasion for the case of concrete treated with solvent-free (neat) silane. This does not hold true in all cases when ordinary silane is applied.

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1 GENERAL

The testing program was selected to assess the effect of mix design, surface finish, and curing/cleaning procedures on the performance of treated concrete. Performance was evaluated using alternate laboratory testing procedures to attempt to examine the tests' advantages and/or limitations in product screening and prediction of field performance. Two basic series of tests were used in various portions of the study: a test series specified by ODOT, and tests based on the Series II procedure of the NCHRP 244 Report. The experimental program is outlined in the following sections. A brief overview of the phases of the testing program is presented, followed by a discussion of the test procedures and materials used in the study.

3.2 OVERVIEW OF TEST PROGRAM

3.2.1 Phase I - Concrete Mix Design

Three types of concrete were investigated, designated classes A, AA, and HD (high density overlay). Class A concrete ($w/c=0.49$) is similar to laboratory reference concrete used in many of the current standardized testing procedures. Class AA concrete has a slightly lower w/c ratio (0.44) and higher 28 day compressive strength than class A. Class HD is used for repairs and overlays of bridge decks, and has a considerably lower w/c ratio (0.33) than concrete classes A and AA.

The effect of mix design on absorption, vapor permeability, depth of penetration, and chloride ion permeability of treated and untreated concrete was studied. Each mix was evaluated using the ODOT test series and the NCHRP 244 Series II procedure. A modification of the NCHRP 244 procedure was also performed to examine the feasibility of shortening the time period required to conduct the basic NCHRP 244 test.

3.2.2 Phase II - Surface Finish

Many standard laboratory tests utilize specimens with either a smooth or broom finish, while a tine finish is often used on bridge decks. Tests were conducted to determine the effect of a tine finish on the performance of treated and untreated concrete. Two mixes, class AA and class HD, were evaluated using the ODOT test series. Performance parameters evaluated included

absorption, depth of penetration, and absorbed chlorides. Results of the tests were compared to those of companion specimens tested in Phase I, which received broom or smooth finishes.

3.2.3 Phase III - Curing and Cleaning Methods

Standard laboratory tests typically utilize wet or moist curing for specimen preparation. However, curing compounds are often used on bridge decks, and these compounds should be removed prior to application of silane. This portion of the study was conducted to evaluate, in the laboratory, certain field curing and cleaning procedures and their effects on the performance of treated concrete.

Three curing methods were examined: 1) use of a linseed oil emulsion compound, 2) use of a pigmented membrane curing compound, and 3) self-curing. Curing compounds were applied directly to freshly cast test specimens. Procedures evaluated to clean the compounds from the concrete included dry shot-blasting, high pressure water washing, and no cleaning. All tests were conducted on class AA concrete and primarily utilized the NCHRP 244 Series II test procedure, although some pilot tests were conducted using a portion of the ODOT test series. Performance parameters examined included depth of penetration and absorption.

3.3 TEST PROCEDURES

A general description of the test procedures used follows. **The majority of the discussion will be presented for the Phase I tests, since this Phase utilized all of the test procedures.** Only deviations from the test procedures will be presented for Phases II and III.

3.3.1 ODOT Series Tests

The ODOT series tests consist of procedures to evaluate depth of penetration, moisture vapor permeability, absorption, and chloride ion intrusion. For all ODOT tests except chloride ion intrusion, specimens were broom finished 203 x 203 x 50 mm (8 x 8 x 2 in.) blocks. The chloride ion intrusion test (salt ponding) utilized 305 x 305 x 76 mm (12 x 12 x 3 in.) blocks.

3.3.1.1 Depth of Penetration, OHD-L40 (ODOT undated, a). One specimen per batch was used for this test. After one week of moist curing, the specimen was oven dried to constant weight and allowed to cool to room temperature. Silane was then applied to the top surface and allowed to cure for a minimum of three days. The block was then broken into four pieces, the pieces wetted, and the depth of the hydrophobic layer was measured at random locations. The

specification requires measurement at 10 locations; however, in this study 12 random readings were taken and the average of readings recorded as the depth of penetration.

3.3.1.2 Moisture Vapor Permeability, OHD-L35 (ODOT undated, b). This test is intended to determine the extent to which absorbed water in treated concrete may be lost through evaporation. Three blocks were moist cured for one week, oven dried to constant weight, and the dry weight recorded. After cooling to room temperature, the blocks were immersed in de-ionized water for 48 hr. After the soaking period the specimens were surface dried, weighed, and all surfaces treated with silane. The specimens were then weighed and oven dried to constant weight to determine the moisture lost.

3.3.1.3 Absorption, ASTM C 642 (ASTM 1987b). Absorption after 48 hr and 50 day immersion in water was measured for both treated and untreated blocks. Twelve specimens per batch were cast: three each for 48 hr and 50 day absorption, treated and untreated. The blocks were moist cured for 28 days, oven dried to constant weight, and the dry weight recorded.

After cooling to room temperature the top (203 x 203 mm) surface of the designated blocks was treated (6 blocks), the remaining surfaces were covered with paraffin wax, and the specimen weights recorded. The treated specimens were allowed to cure for at least three days before immersion. At the end of the specified immersion period the specimens were surface dried and weighed to determine the amount of water absorbed.

3.3.1.4 Chloride Ion Intrusion, AASHTO T259/T260 (AASHTO 1986b, AASHTO 1986c). Seven blocks were used for each batch: three treated, three untreated, and one control (untreated, unponded). The blocks were moist cured for 14 days, then stored in a climate controlled environmental chamber (23°C, 50% RH). Treatment was applied to the designated specimens after one week of drying in the environmental chamber (at 21 days of age). One week later, dams were placed on all specimens except the control specimen. The dams were made of 19 mm high by 13 mm wide neoprene strips.

Two weeks later the top surface of all specimens (except control) was ponded with a 3% NaCl solution for a period of 90 days. Each ponded specimen was covered with a glass plate to reduce evaporation of solution. A typical ponded specimen is shown in Fig. 3.1. At the conclusion of the 90 day ponding period, the specimens were allowed to dry a minimum of 5 days prior to sampling for chloride analysis. For each specimen, three holes were drilled using a rotary hammer, and powder samples taken at two depth intervals: 1.6 to 13 mm (1/16 to 1/2 in.), and 13 to 25 mm (1/2 to 1 in.), that is, 6 chloride samples were taken from each block. The rotary hammer was mounted on a press as shown in Fig. 3.2, the specimen was clamped to the base of the press, and a scale was used to control the depth of the drilling. Two drilling bits with straight blades were

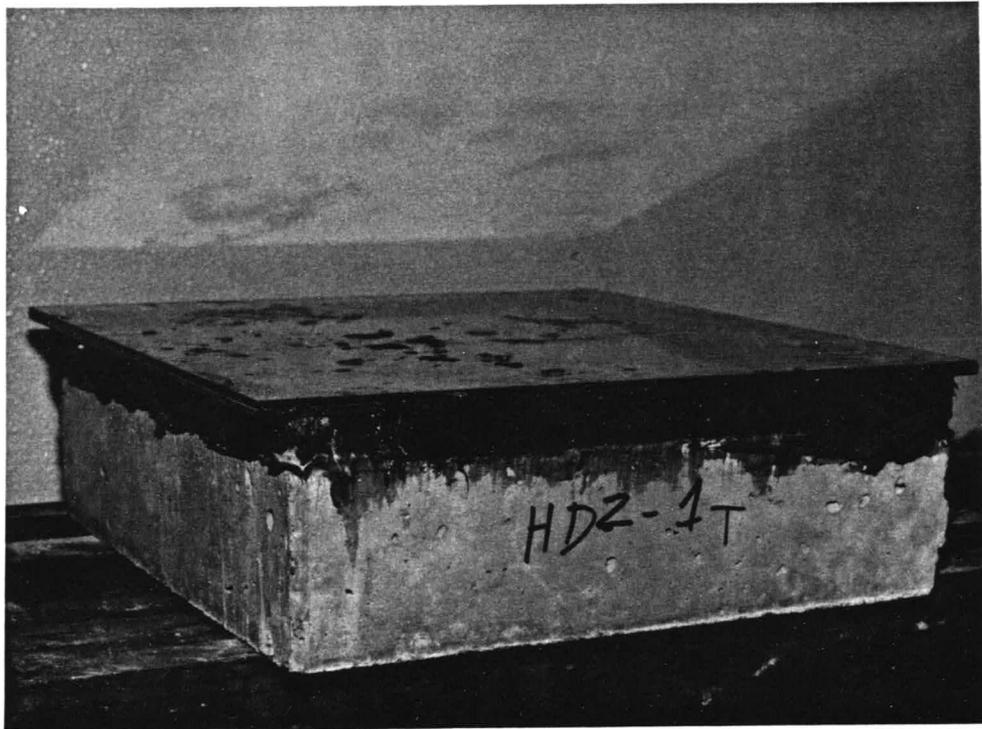


Fig. 3.1 Salt Ponding Specimen

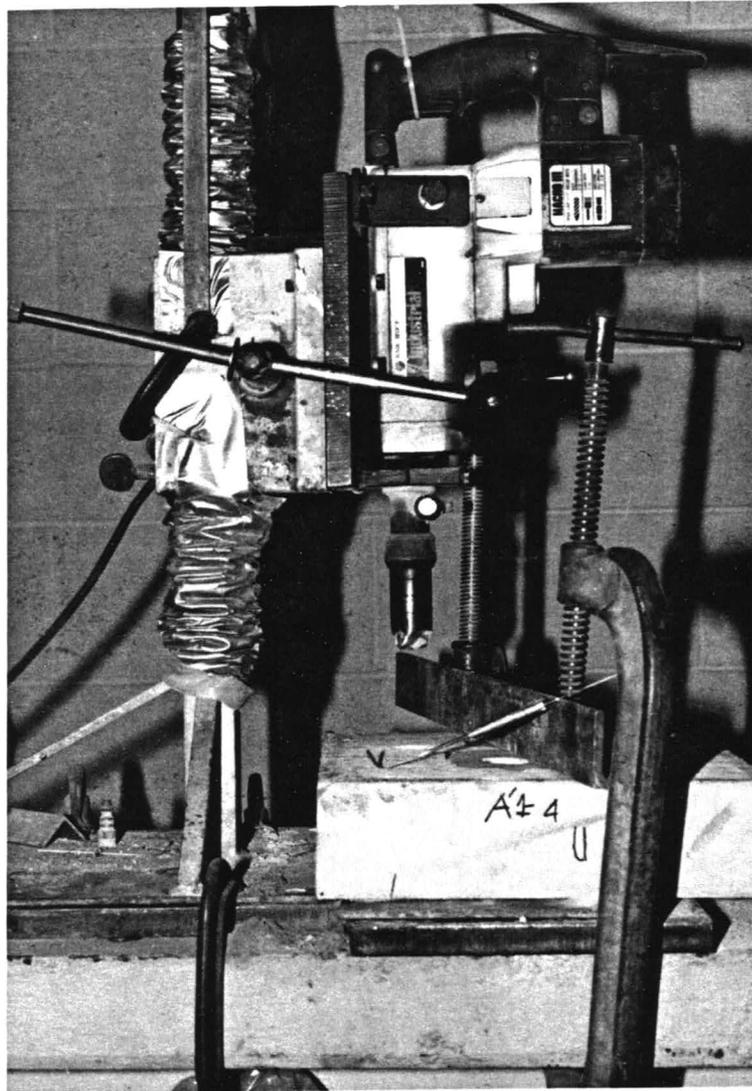


Fig. 3.2 Drilling Setup for Chloride Samples

used. The bits were designed to produce a drilled hole with a flat bottom for accurate depth control. The larger bit was 32 mm (1.25 in.) in diameter, and was used to remove the top 1.6 mm (1/16 in.) layer, and the first depth interval. For the second depth interval a 25 mm (1.0 in.) diameter bit was used. Retrieval of powder was achieved by using a stainless steel spoon. Prior to each drilling stage, the bits and sampling spoon were thoroughly cleaned using isopropyl alcohol to avoid contamination of powder samples.

Chemical analyses were conducted by the University of Oklahoma Chemistry Department. Tests were performed according to AASHTO T 260-84, and results reported as total chlorides absorbed (total chlorides minus total base chlorides contained in the control specimen). To reduce testing costs, chloride analysis was limited to specimens of the first batch of each concrete class.

3.3.2 NCHRP 244 Series II Tests

The basic test procedures described in the NCHRP 244 Report (Pfieffer and Scali 1981), with slight modifications as will be discussed, were used. Two additional variations of the procedures were also examined.

The basic test procedure involved casting 102 mm (4 in.) cubes which were self-cured in plastic bags for one week in the environmental chamber. The cubes were then lightly sand blasted to remove surface laitance, weighed, returned to the plastic bags, and stored in the environmental chamber for two weeks of additional curing. Cubes were then removed from the bags, allowed to dry in the environmental chamber for five days, and treatment was applied to designated specimens. All specimens were weighed; treated specimens were weighed before and after treatment. The cubes were allowed to air dry in the environmental chamber for an additional 26 days, and were weighed at intervals of about 7 days.

The cubes were then immersed in a 15% NaCl solution for 21 days, in plastic containers covered with plastic sheets to reduce evaporation. The solution was maintained at 25 mm (1 in.) above the top surface of the cubes, with weight recordings performed every 3 days. Immersed cubes are shown in Fig. 3.3. At the completion of the immersion period, the cubes were allowed to air dry in the chamber for 21 days, with weights recorded every 3 days.

The minor variations from the basic procedures of the NCHRP Series II test are as follows. For each batch, triads of cubes rather than pairs of cubes as used in the NCHRP study, were used for each test group. This was done to facilitate statistical comparisons with the results of ODOT series tests. Chloride samples taken at the conclusion of the test procedure were obtained by rotary hammer drilling into three surfaces of the cube at two depth intervals, similar to AASHTO T259/T260 procedures. This differs from the original 244 report, where samples were obtained by

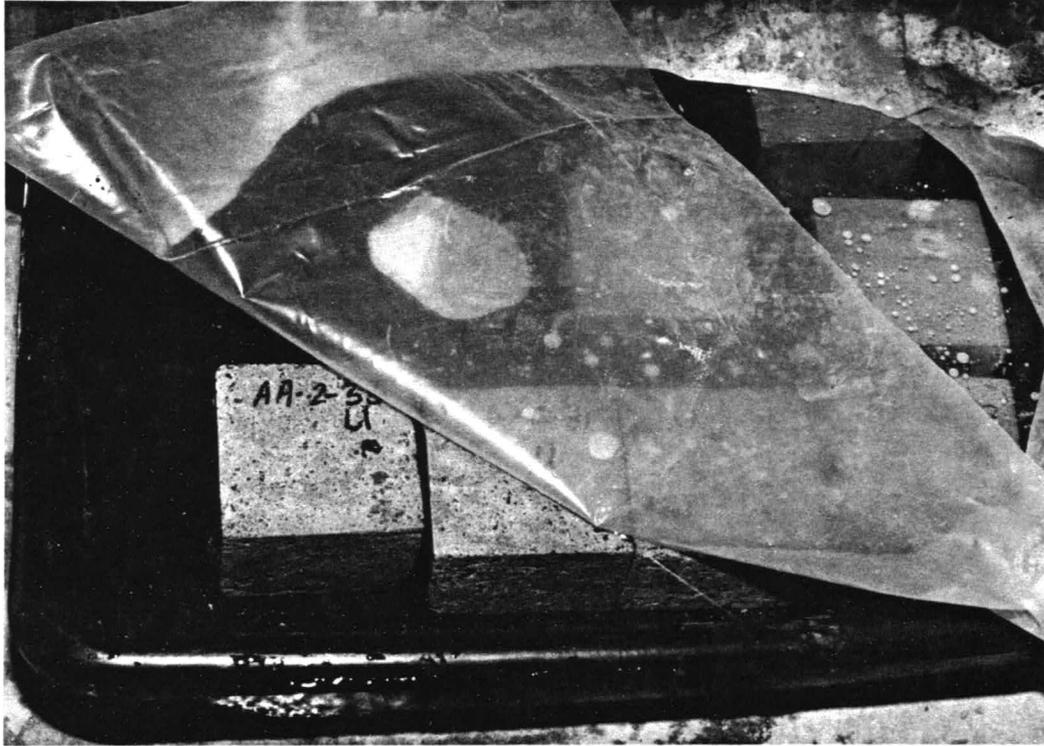


Fig. 3.3 Immersed Cube Specimens

crushing one half of a cube to a fineness sufficient for chloride analysis. Reasons for using drilled powder samples, rather than crushing, will be presented in the discussion of test results (Chapter 4). The same drilling assembly and powder sampling procedures described in the salt ponding test were used for the cubes.

In parallel with the basic test procedure described above, two additional variations of the NCHRP 244 cube tests were examined. One triad of treated cubes was tested following the basic procedure, with the exception that the cubes were not sandblasted prior to treatment. The second additional procedure utilized triads of treated and untreated cubes, and differed from the basic procedure only in that the cubes were oven dried, rather than air dried, at the conclusion of the 21 day immersion period. This series was conducted with the intent of reducing the overall time period required for the test procedure. In summary, 15 cubes were cast for each batch: 6 for the basic procedure (3 treated, 3 untreated), 6 for the modified procedure utilizing oven drying (3 treated, 3 untreated), and 3 treated cubes which were not sandblasted. Chloride analysis was performed only for the cubes which followed the basic test procedure, for the first batch of each mix type.

3.3.3 Other Depth of Penetration Tests

Concrete moisture content, among other factors, influences the depth of penetration of silane water repellent treatment materials. The various tests performed utilized a wide range of moisture contents at treatment (from oven dry to nearly saturated surface dry), so depth of penetration was measured for selected specimens (in addition to the primary purpose of the original test). Measurements were taken using absorption specimens, cubes, and moisture vapor permeability (MVP) specimens. A pilot study was conducted to examine the effect of retreatment on specimens which initially exhibited negligible penetration. Previously untreated specimens from earlier tests were treated at random moisture contents to examine the effect on penetration. A controlled study to examine the effect of moisture content on penetration was also performed using mix type AA.

3.3.4 Phase II Tests

Tests utilized the ODOT series procedures, since the test surface is always the top surface of the specimen and a tine finish could be easily applied to the fresh concrete. A steel rake was used to create the tine finish. Exceptions to the procedures previously discussed are noted below.

1. Absorption, ASTM C642. Only six specimens per batch were used: three treated and three untreated. The same specimens were used to obtain both 48 hr and 50 day absorption.

2. Chloride Ion Intrusion, AASHTO T259/T260. Due to the low slump range of the class HD mix, specimens were cast in two batches, so two control specimens were cast (one for each batch). Thus, a total of 8 specimens was used.

3.3.5 Phase III Tests

Primary testing to evaluate curing/cleaning procedures followed the NCHRP 244 test. A pilot study was also conducted to examine curing/cleaning for a tine finished surface. The pilot study was carried out using the ODOT series tests.

3.3.5.1 NCHRP 244 Series II. Three curing methods and two cleaning methods prior to treatment were tested. The combination of variables included: 1) linseed oil curing (uncleaned, shot-blasted, high pressure water washed), 2) use of a pigmented membrane curing compound (uncleaned, shot-blasted, high pressure water washed), and 3) self-curing (shot-blasted, high pressure water washed). A total of 24 cubes was cast (3 for each of the 8 conditions noted), using mix class AA.

Certain variations from the basic procedure were necessary to accommodate the curing and cleaning procedures. The sand-blasting step to remove surface laitance was omitted in favor of either shot-blasting or high pressure water washing, as appropriate. When a curing compound was used, the compound was applied to the top surface of the freshly cast cube immediately after the superficial water subsided. The compound was applied to the remaining five sides of the cube on the following day when forms were removed. Self-cured cubes were placed in plastic bags as before, and all cubes stored in the climate controlled chamber for curing. All cubes were cured for 21 days, and cleaned by the designated methods at 21 days age. No chloride analysis was performed on the specimens.

3.3.5.2 ODOT Series. A pilot study was conducted to examine the effect of linseed oil curing for a tine finish. These results could be compared to those of Phase II, which utilized moist cured, tine finished specimens. Only depth of penetration and absorption were examined.

Two specimens were used to examine depth of penetration. Linseed oil emulsion was applied as described above for the cube specimens. After 7 days of curing, the top surface of one specimen was cleaned by shot-blasting, and the other remained uncleaned. The remainder of the test followed the standard ODOT procedure.

Six specimens were tested for absorption following ASTM C642. After curing for 28 days, three were shot-blasted and three remained uncleaned. The remainder of the test followed the standard procedure.

3.4 MATERIALS AND SPECIMEN PREPARATION

3.4.1 Silane

The silane weatherproofing material used in all phases of the research was produced by the Chemistry Department of the University of Oklahoma, to ensure quality and reproducibility of the material. The solution contained 40.3% (by weight) isobutyltrimethoxysilane in an isopropyl alcohol carrier, with a recommended application rate of 3.07 m²/L (125 ft²/gallon). The silane was applied using an equivalent application rate of 270.2 gm/m² (25.1 gm/ft²). Silane was brushed onto each treated surface and controlled by weighing the silane and using a saturated brush at the start of application. After 12 hr, samples were sprayed with a light water mist and allowed to cure an additional three days (minimum) prior to testing. The infrared spectra of the silane material used is shown in Fig. 3.4.

3.4.2 Phase I

For each mix type, two batches were cast with the same mix design. Specifications for the mixes, as well as actual mix properties, are shown in Table 3.1. Batches of concrete types A and AA were mixed in a single loading of the mixer; the size of each batch was 0.127 m³ (4.5 ft³). Batches of the high density concrete were cast in the same mixing session, but due to the mix's low slump range, each batch was divided into equal half-batches to facilitate proper mixing and specimen fabrication. For each mix three cylinders were cast and tested in compression at 28 days of age. The reported strengths are averages of 3 cylinders for each batch (each point represent the average of 6 tests), except for class HD, where 6 cylinders per batch were tested (average of 12 cylinders).

Several trial mixes were batched to reach the final mix design for each concrete class. The final proportions of the mixes were maintained within each concrete class. The w/c ratios listed in Table 3.1 were accurately maintained for all batches.

The basic variables used to produce concrete with the required specifications were the coarse gravel to fine aggregate ratio, and dosage of air entraining admixture (Neutralized Vinsol Resin). In the case of class HD concrete, a superplasticizer was also employed to attain an acceptable range of slump. Due to relatively high ambient temperatures during casting of certain batches, somewhat increased air entraining admixture dosages were required to obtain the specified air contents. Difficulties were encountered for class AA batches in obtaining the specified air content with the recommended admixture dosages. For this reason, air contents of class AA batches were slightly below the specified range (Table 3.1). The admixture dosages were increased

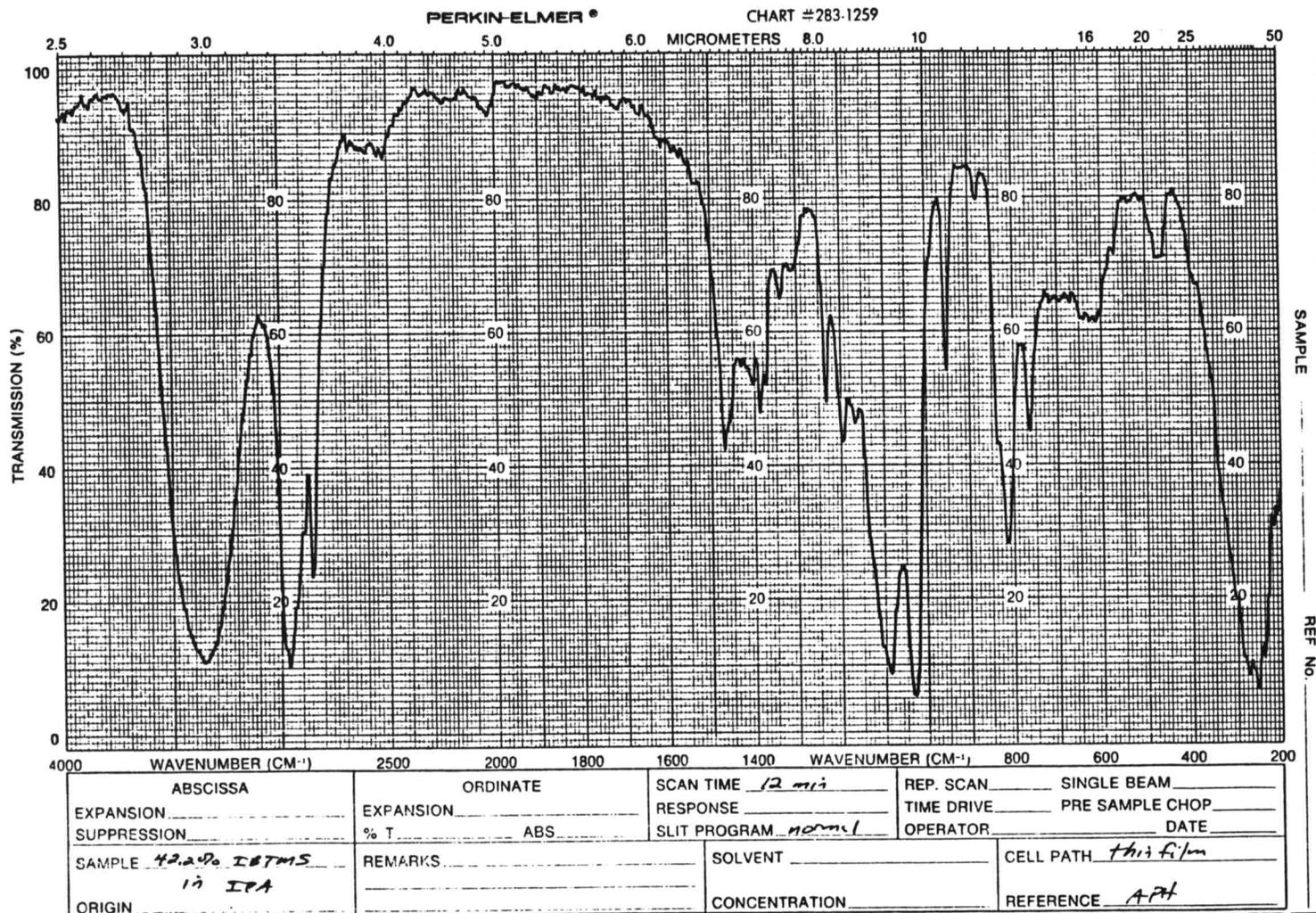


Fig. 3.4 Infrared Spectra of Silane Material

Table 3.1 Specifications and Measured Properties of Mixes (Phase I)

| Mix Type | A | AA | HD |
|---|---------|---------|---------|
| W/C Ratio | 0.49 | 0.44 | 0.33 |
| Cement Factor (kg/m ³) | 335 | 390 | 490 |
| Specified Slump (mm) | 25-75 | 25-75 | 13-25 |
| Specified Air Content (%) | 5-7 | 5-7 | 5.5-7.5 |
| Measured Slump* (mm) | 50, 50 | 38, 25 | 13, 19 |
| Measured Air Content* (%) | 6.5,5.2 | 4.9,4.5 | 5.2,6.0 |
| Maximum Size Aggregate+ (mm) | 13 | 13 | 13 |
| Coarse Aggregate** (kg/m ³) | 950 | 950 | 964 |
| Fine Aggregate** (kg/m ³) | 869 | 802 | 640 |
| 28 Day Compressive Strength (Mpa) | 39.3 | 46.2 | 56.5 |

(1 lb/cyd = 1.68 kg/m³, 1 mm = 0.039 in, 1 Mpa = 0.145 ksi)

+ Maximum aggregate size used in laboratory concrete specimens

* Values for each of two batches

** Batch weights at saturated surface dry condition

in subsequent batches. Typical air entraining admixture dosages (cc/Kg of cement) were 1.4, 0.93, and 2.0 for classes A, AA, and HD, respectively.

Three types of specimens were prepared for each concrete class. In all cases wood forms were used, and the surfaces exposed to concrete were treated with clear acrylic coating to avoid deterioration of forms and to avoid using form oil or other materials which may interfere chemically with the tested material. All specimens were covered with wet burlap and plastic sheets for 24 hr of initial curing.

For the ODOT series, 203 x 203 x 50 mm (8 x 8 x 2 in.) specimens were cast in pairs of blocks in each form as shown in the middle portion of Fig. 3.5. The top surface of all the ODOT series test specimens was broom finished. The salt ponding test specimens (AASHTO T259) were 305 x 305 x 75 mm (12 x 12 x 3 in.) slabs. Each specimen was cast in a single form as shown in the upper portion of Fig. 3.5, and the top surface trowel finished. For the NCHRP 244 series II tests,

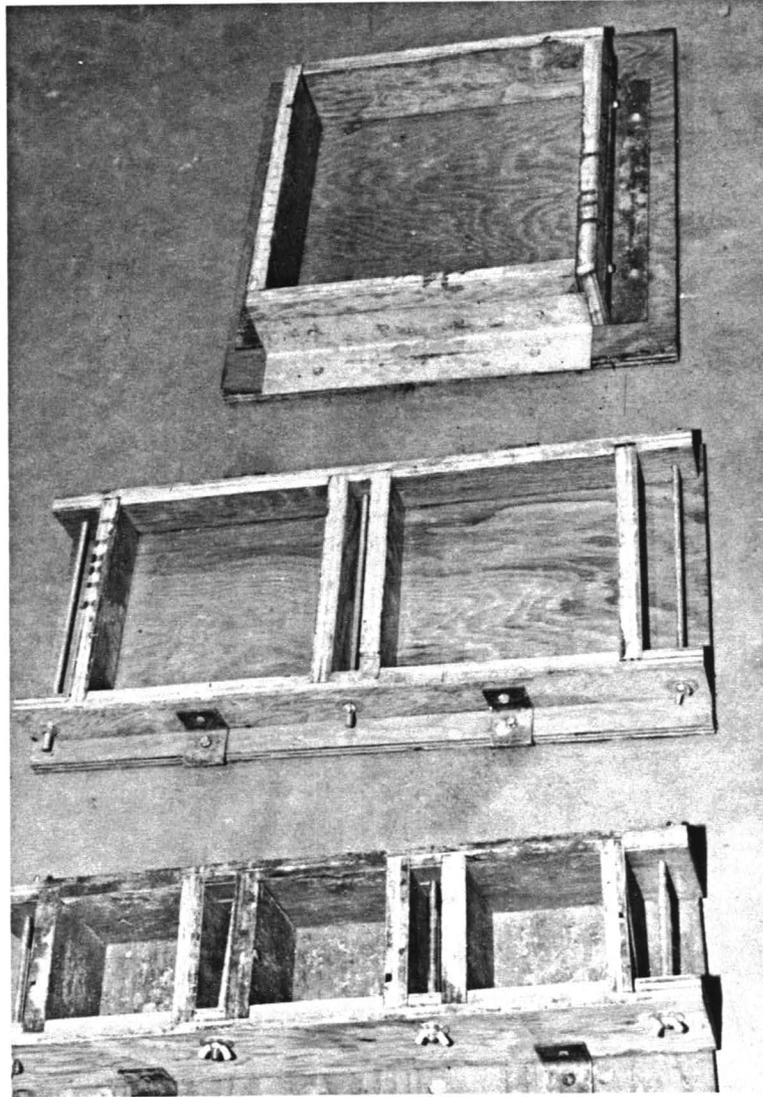


Fig. 3.5 Forms Used for Specimens

102 mm (4 in.) cubes were cast in gang forms of three cubes as shown in the lower portion of Fig. 3.5 and in Fig. 3.6. All specimens were trowel finished.

Moist curing for ODOT series specimens was performed by placing the specimens in lime saturated water after the initial 24 hr curing. Galvanized steel tanks were used as curing tanks; a heater (with thermostat) and a submersible pump were placed in each tank to maintain uniform temperature and circulation. At designated stages in the testing procedures, specimens were moved into the climate controlled chamber and stored on racks as shown in Fig. 3.7. NCHRP Series II test specimens were cured in plastic bags (immediately after removal from the forms) and stored in the controlled climate chamber. The specimens were kept in the chamber throughout the test procedure. (Note: The cube specimens seen in Fig. 3.7 were used in Phase III, and were not cured in plastic bags.)

3.4.3 Phase II

Batching, casting, and curing procedures were the same as for Phase I, except that tine finishes were applied to specimens. The rake used to produce the tined surface was fabricated to produce grooves 3 mm (1/8 in.) wide, 4.8 mm (3/16 in.) deep, and spaced 19 mm (3/4 in.) on center. One batch each of mix classes AA and HD were cast, with the class HD mix cast in two half-batches. Mix proportions used were the same as for Phase I. A typical tine finish for a class HD specimen can be seen in Fig. 3.8. Measured slumps and air contents were 70 mm (2.75 in.) and 5.0% for class AA, and 16 mm (5/8 in.) and 5.5% for class HD. Compressive strengths at 25 days were 45.4 Mpa (6.6 ksi) and 62.3 Mpa (9.0 ksi) for mix classes AA and HD, respectively.

3.4.4 Phase III

Class AA concrete was used, and specimens were cast in two batches (one batch for NCHRP 244 cube tests, one batch for ODOT tests). Measured slumps and air contents were 51 mm (2 in.) and 6.0% for the NCHRP 244 cube specimens, and 46 mm (1.75 in.) and 6.4% for the ODOT slab specimens. Compressive strengths at 28 days were 42.8 MPa (6.2 ksi) and 40.7 MPa (5.9 ksi), respectively.

Sealtight lin-seal penetrating concrete sealer, manufactured by W. R. Meadows, Inc., P.O. Box 543, Elgin, IL 60121, was used for the linseed curing compound. The compound is a high solids solution of boiled linseed oil. The recommended application rate of 7.37 m²/L (300 ft²/gal) was used. Sealtight 1600-white series water-base pigmented concrete curing compound, from the same manufacturer, was used at the recommended application rate of 4.9 m²/L (200 ft²/gal). These



Fig. 3.6 Specimen Casting, NCHRP 244 Cubes



Fig. 3.7 Controlled Climate Chamber

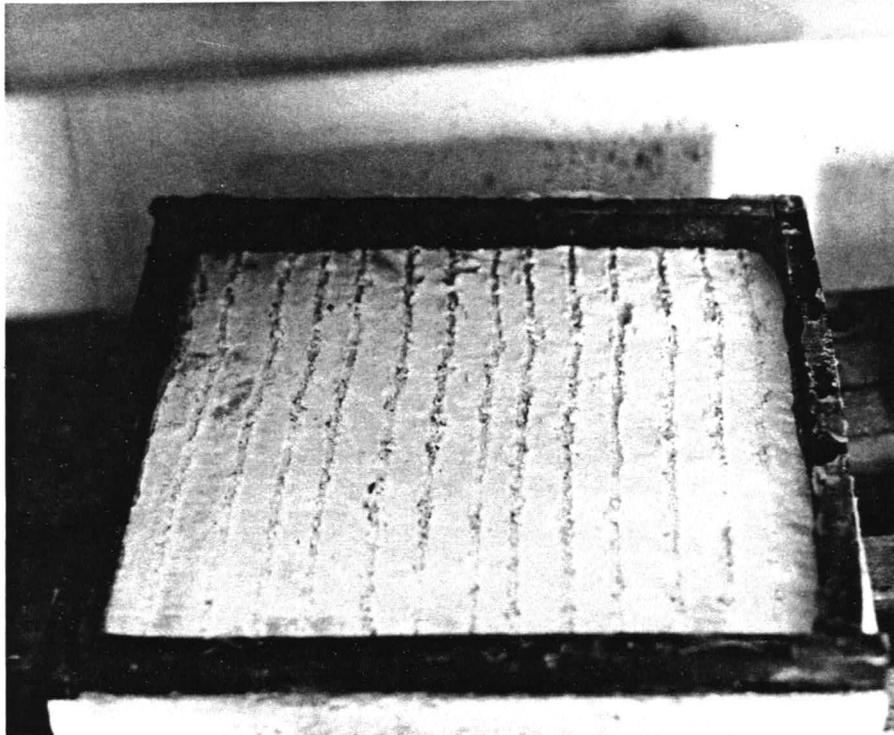
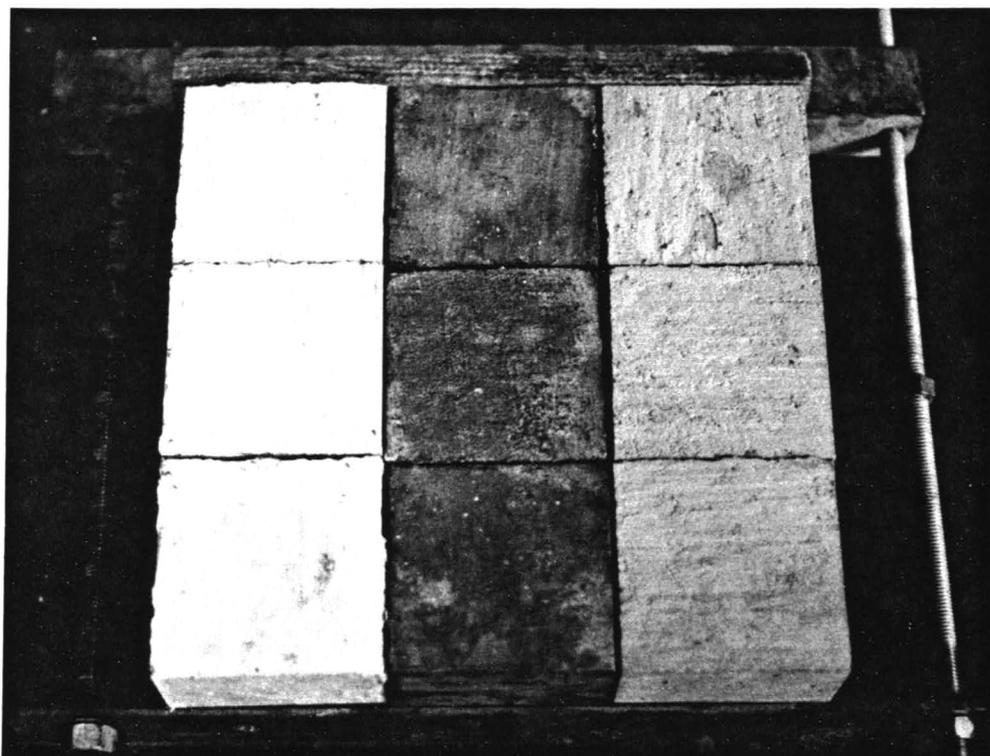


Fig. 3.8 Tine Finish Class HD Specimen

compounds were hand-sprayed onto the top surface of freshly cast test specimens, and brushed onto the remaining surfaces immediately after removal from the forms. Cube specimens with applied curing compounds can be seen in the photo of Fig. 3.9.

Shot-blasting was performed using S-280 steel shot. Blasting proceeded until the cleaning compounds were no longer visible. The resulting surface finish is shown in Fig. 3.10. High-pressure water washing was performed using a 17 Lpm (4.5 gpm) pump which delivered 62° C, 20 MPa (170° F, 2900 psi) water at the nozzle. A 15° wand was held approximately 75 mm (3 in.) above the specimen surface, and two passes were made in order to remove the curing compounds, as shown in Fig. 3.11. The resulting surface finish was similar to that of the shot-blasted cubes.



(Left: Pigmented Membrane, Center: Linseed Oil, Right: No Curing Compound)

Fig. 3.9 Phase III Cube Specimens Prior to Cleaning



Fig. 3.10 Phase III Cubes After Shot-Blasting



Fig. 3.11 High Pressure Water Washing, Phase III Cubes

CHAPTER 4

RESULTS OF PHASE I - CONCRETE MIX DESIGN

4.1 INTRODUCTION

Results are presented to give insight into the four basic behavioral parameters studied, namely: depth of penetration, water absorption, chloride ingress, and moisture vapor permeability. Test results are presented according to the parameter measured. All results presented in this chapter are averages of individual test specimens for each mix type. Certain individual specimen results are presented in Appendix A.

4.2 DEPTH OF PENETRATION

Primary depth of penetration results were obtained from the ODOT test, OHD-L40 (ODOT undated, a). Additional depth of penetration results were obtained from selected specimens at the conclusion of other tests. Specimens from NCHRP 244 Series II tests, absorption tests, and moisture vapor permeability (MVP) tests were examined for depth of penetration. Specimens for the depth of penetration and absorption tests were treated with silane after oven drying (zero moisture content), while the MVP specimens were originally treated at a nearly saturated surface dry state. The high moisture content of the MVP specimens resulted in negligible penetration depth for all mix types. To examine the feasibility of retreatment, the MVP specimens were oven dried, and silane reapplied at the recommended coverage rate ($3.07 \text{ m}^2/\text{L}$ ($125 \text{ ft}^2/\text{gal}$)) and half that rate ($6.14 \text{ m}^2/\text{L}$ ($250 \text{ ft}^2/\text{gal}$)).

Depth of penetration results for all mix types are shown in Table 4.1. For each batch one depth of penetration specimen, two moisture vapor permeability specimens, and one absorption specimen was used. Silane penetration was observed to be fairly uniform in all dry application specimens, within the mix type and within each specimen.

Silane penetration was consistently higher for class HD concrete. This could possibly be due to mix configuration, pore size, and higher capillary suction forces. Mix type A ($w/c = 0.49$) yielded higher depth of penetration values than type AA ($w/c = 0.44$). Thus, results of these tests suggest that the w/c ratio alone does not provide a relative indication of performance.

It should be noted that although the depth of penetration is an important factor in assessing the performance of silane, its effect on other performance characteristics is unknown. Water

Table 4.1 Depth of Penetration Results for Dry Application (mm)

| Mix Type | Depth of Penetration Test | Absorption Specimens | MVP Specimens, 1/2 Rate Reapplied | MVP Specimens, Full Rate Reapplied |
|----------|---------------------------|----------------------|-----------------------------------|------------------------------------|
| A | 11.9 | 11.7 | 9.1 | 11.9 |
| AA | 9.4 | 8.6 | 8.4 | 11.9 |
| HD | 13.5 | 15.5 | 10.2 | 14.5 |

(1 mm = 0.039 in)

sprayed on the surface of originally treated MVP specimens formed beads, which indicates surface hydrophobicity even though no measurable penetration existed.

It can be seen that retreatment of specimens which originally had no measurable penetration lead to significant improvement. Retreatment at full coverage rate resulted in penetration comparable to the other dry treated specimens. It should be recognized that the retreated specimens were oven dried prior to reapplication of silane, and the scope of this portion of the study was very limited.

Concrete moisture content is known to affect silane penetration. An example is shown in Fig. 4.1, where the depth of the hydrophobic layer can be seen for specimens from the same mix and batch, but treated at varying moisture contents (the hydrophobic layer has been delineated by black lines in the figure). A preliminary examination of the effect of moisture content on the depth of penetration of silane was carried out using surplus and/or originally untreated specimens from the absorption test and the treated NCHRP 244 cubes. Selected absorption test specimens were allowed to air dry and were treated at various in-situ moisture contents. No attempt was made to control the moisture content of the specimens. Due to various factors, the moisture contents could be determined only approximately. Moisture contents at the time of treatment for NCHRP 244 cubes were estimated based on the oven dry weight obtained at the end of the 244 test. The oven drying process is expected to remove the moisture but the absorbed salt and the weight of silane remaining could not be accurately estimated.

Results shown in Fig. 4.2 indicate that concrete moisture content at time of treatment can have a dramatic impact on the depth of penetration, and the effect of moisture content on silane

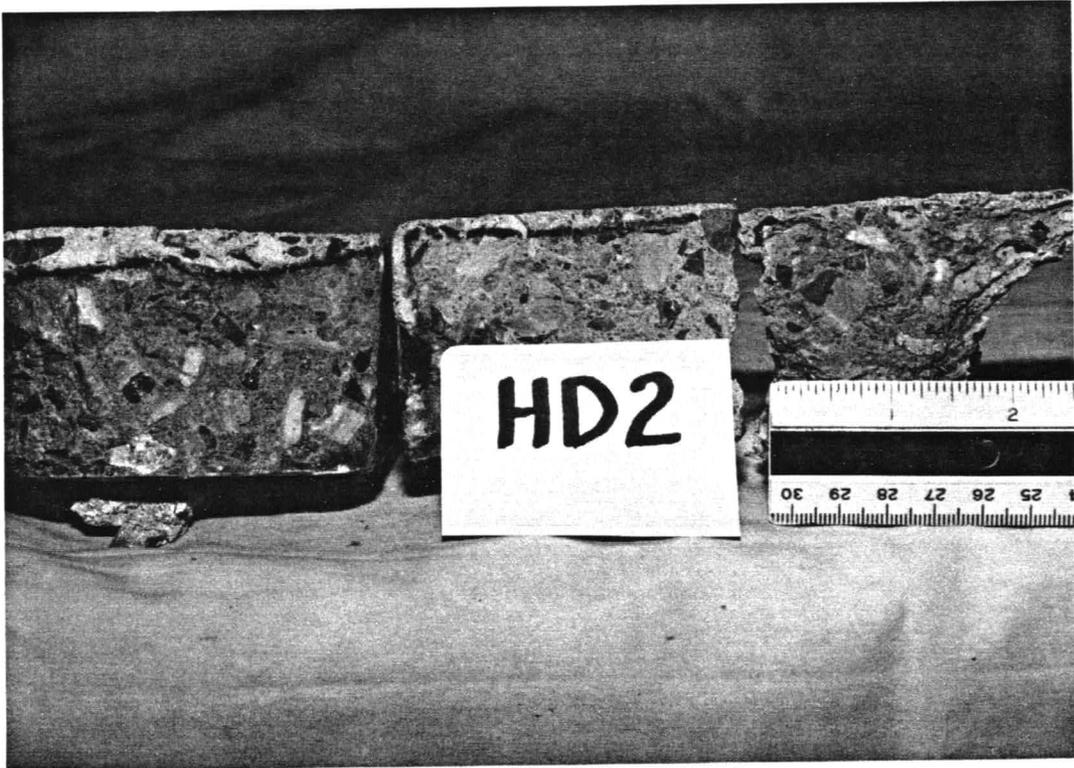


Fig. 4.1 Penetration for Class HD Specimens: (L-R) Dry, Moist, Saturated at Treatment

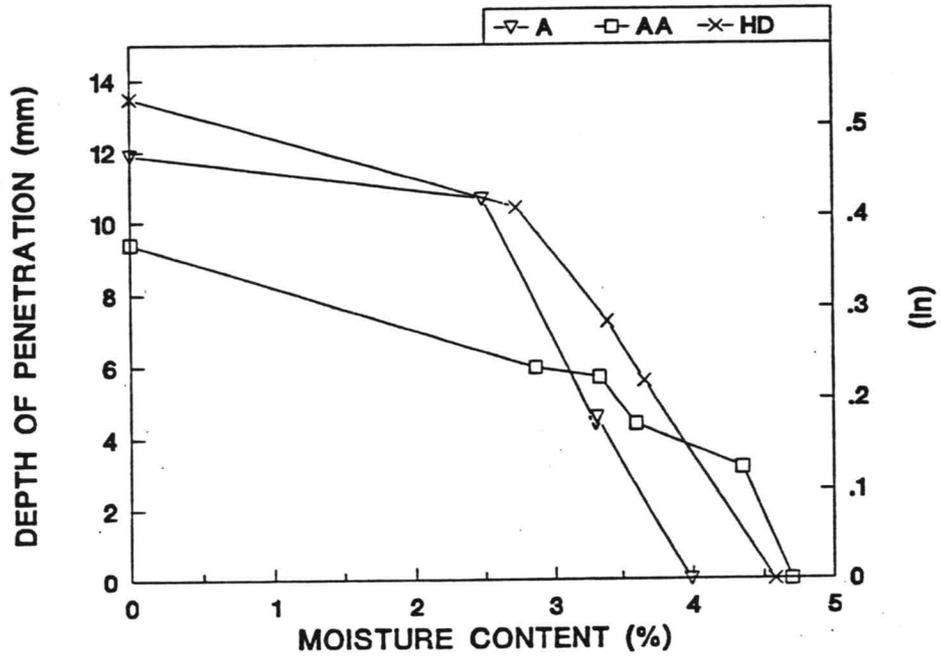


Fig. 4.2 Influence of Moisture Content on Penetration, Pilot Study

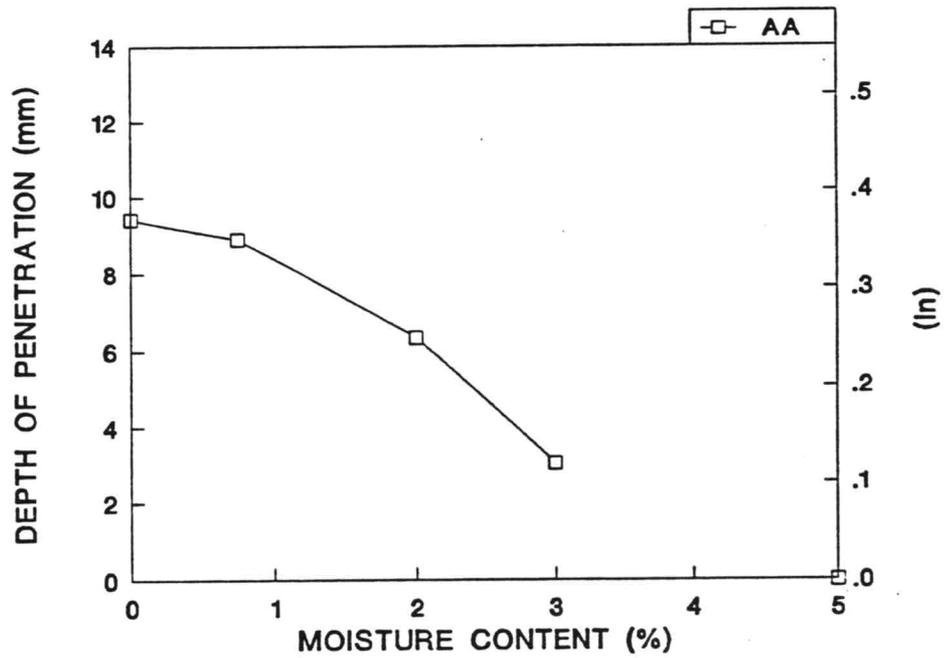


Fig. 4.3 Influence of Moisture Content on Penetration, Controlled Study

penetration appears to be affected by the mix type. For moisture contents over 2% the penetration of silane could be significantly reduced.

A controlled study of the effect of moisture content at time of treatment on silane penetration was performed for class AA concrete. Specimens were treated at target moisture contents and the depth of penetration measured. Results of the test, shown in Fig. 4.3, indicate that penetration may begin to be affected at moisture contents as low as 1%.

4.3 WATER ABSORPTION

Water absorption characteristics of treated and untreated specimens were obtained from two tests. The ODOT test is designed specifically for this purpose, and the weight gained by cubes immersed in a 15% NaCl solution is used as an indicator of absorption for the NCHRP 244 test.

4.3.1 ODOT Series Test

Absorption results, obtained from ODOT tests following ASTM C 642, are shown in Table 4.2 and Fig. 4.4. Concrete class A absorbed slightly more water than class AA, even though class A specimens had a higher depth of penetration. In general, class HD concrete absorbed the least amount of water, except for the case of treated specimens after 48 hr of immersion.

Table 4.2 Results of ODOT Series Absorption Tests

| Concrete Class | | A | AA | HD |
|-----------------------------|-------------|------|------|------|
| 48 hr Absorption (%) | Untreated | 4.28 | 4.12 | 3.98 |
| | Treated | 0.55 | 0.17 | 0.36 |
| | Improvement | 87% | 96% | 91% |
| 50 day Absorption (%) | Untreated | 5.25 | 5.11 | 4.68 |
| | Treated | 2.20 | 0.79 | 0.77 |
| | Improvement | 58% | 85% | 84% |

Improvement due to treatment can be seen in Fig. 4.5. The improvement due to treatment was higher for concrete class AA than for the other two concrete classes for both 48 hr and 50 day absorption. Generally, for all concrete classes the percent improvement due to treatment dropped

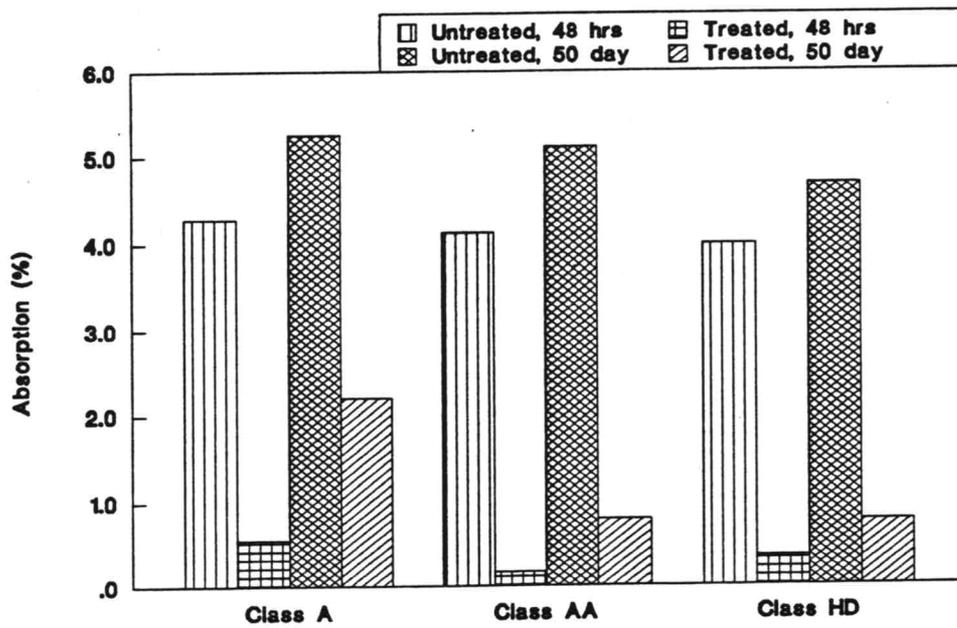


Fig. 4.4 Absorption Results, ODOT Series Test

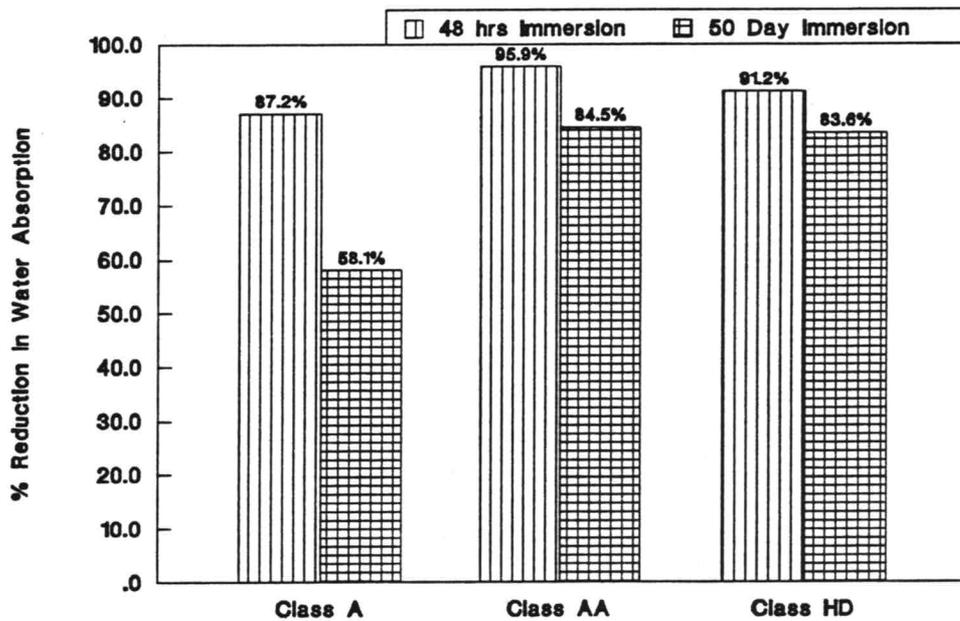


Fig. 4.5 Improvement in Water Absorption Due to Treatment, ODOT Absorption Test

after 50 days, which reflects the difference in absorption rates between untreated and treated specimens. There is no indication that the decrease in the improvement is due to a deterioration of the sealer; rather it seems that the treated specimens absorbed moisture at a much slower rate than the untreated specimens. For the untreated specimens, approximately 80% of the 50 day moisture gain was absorbed during the first 48 hr of soaking, whereas the treated specimens absorbed only 20-45% of the 50 day moisture gain during the first 48 hr of immersion. This is a strong reminder of the importance of recognizing the assumptions used in the testing procedures in assessing the possible benefits of treatment. The duration of immersion is a significant factor.

For this series of testing the general trend was the higher the w/c ratio, the more water absorbed, a trend consistent with the basic doctrines in concrete mix design. This, however, is not so clearly projected for the NCHRP 244 series tests as will be seen next.

4.3.2 NCHRP 244 Series II Test

Weight gained upon immersion in a 15% NaCl solution was monitored over the 21 day soaking period. Also, weight loss during 21 days of air drying (upon removal from the solution) was recorded. Weight gained/lost was normalized with respect to the cube weight immediately prior to immersion, since dry weight of the cube is not obtained from the test procedure. It must be noted that the weight gained in this test includes the weight of chlorides absorbed by the specimens. In this discussion, however, it will be assumed that inferences regarding water absorption characteristics can be made with an acceptable degree of accuracy.

Approximate moisture contents at time of treatment and immersion were obtained from the cubes which underwent oven, rather than, air drying. The oven dry weight included the weight of absorbed salt and, for treated specimens, the weight of silane. These approximate moisture contents at treatment were 3.3, 4.0, and 3.5 percent for mix types A, AA, and HD, respectively.

The moisture contents at the end of the immersion period were used to perform a series of two-way hypothesis tests using the t-statistic. All data for sand blasted cubes data were used in the statistical study, regardless of the drying conditions (air or oven drying). This allowed the use of a larger number of data points, that is 12 data points for each concrete class. The statistical analysis of the data confirmed that the differences in the means (% weight change at 21 day of immersion) were significant at the 5% level between all mixes and treatment conditions, except for treated concrete classes A and AA, where there was no statistical difference. A summary of the averages and standard deviations is contained Table 4.3.

Average weight gained/lost for 6 cubes of each concrete class (3 per batch) is presented for untreated specimens in Fig. 4.6, and for treated cubes in Fig. 4.7. Untreated class AA

Table 4.3 Means and Standard Deviations for NCHRP Water Absorption Data
(% Moisture Content)

| Concrete Class | Mean | Std. Deviation |
|----------------|------|----------------|
| A Treated | 0.45 | 0.062 |
| A Untreated | 0.80 | 0.069 |
| AA Treated | 0.46 | 0.085 |
| AA Untreated | 1.11 | 0.114 |
| HD Treated | 0.26 | 0.018 |
| HD Untreated | 0.46 | 0.022 |

specimens gained more weight upon immersion than the other two concrete classes. This trend was not significantly reflected for treated specimens, where the weight gained by class A and class AA specimens was very similar. This result is unexpected considering the higher w/c ratio for concrete class A as compared to class AA. It is possible that the mix proportions, and the moisture content of the specimens at immersion and treatment lead to the increased absorption of water and, as will be discussed later, chlorides for class AA. As can be seen in Figs. 4.6 and 4.7, compared to the other two mix types the treated and untreated type HD specimens gained the least amount of weight upon immersion. Also, untreated HD cubes performed as well as treated A and AA cubes. Comparing the performance of treated and untreated concrete (Figs. 4.6 and 4.7), it can be seen that after treatment the specimens of all classes gained weight at a much slower rate, and that the maximum weight gain was significantly reduced.

The improvement values for the NCHRP cubes shown in Fig. 4.8 (determined at 21 days of immersion) indicate that in general, the improvement due to treatment was less than for companion specimens tested using the ODOT procedures. It must be noted that the ODOT series specimens were immersed in water rather than in 15% NaCl solution. Also, silane was applied to concrete with moisture contents from approximately 3.3 to 4% for the NCHRP test, rather than to oven dried specimens as for the ODOT absorption tests (Table 4.2, Fig. 4.5).

Again, class AA benefitted most from treatment, a trend consistent with previous observations in the ODOT series absorption test. Also, the improvement due to treatment decreased with time for cube specimens. A useful way to examine this trend is to consider the behavior shown in Fig. 4.9, where the reduction in weight gain can be seen throughout the immersion period. It would seem that a sustained decline in improvement can be detected. Due to the limited period of immersion it is not clear whether this decline in improvement will continue.

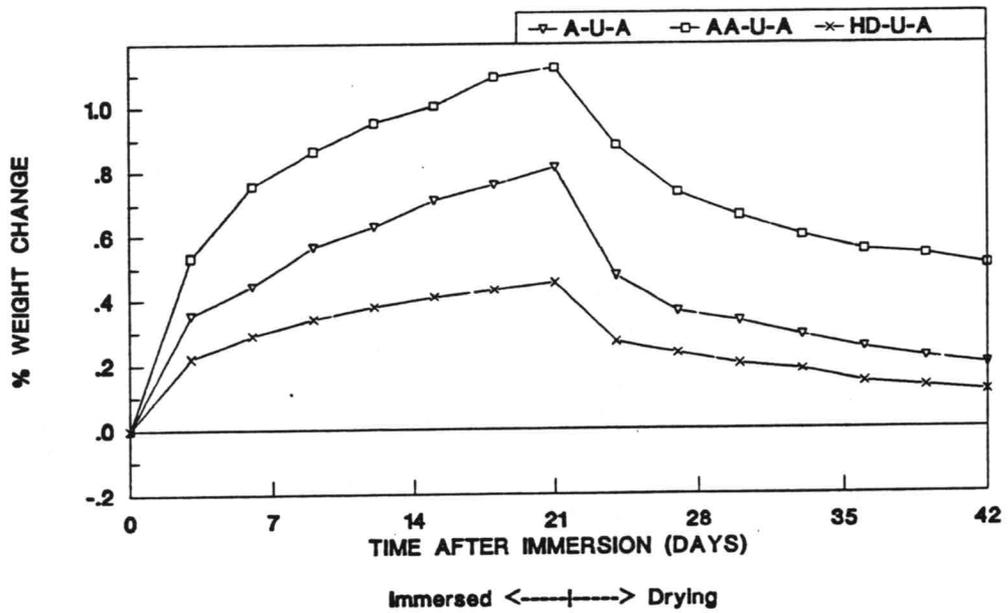


Fig. 4.6 Weight Gain/Loss of Untreated Cubes (Air dried)

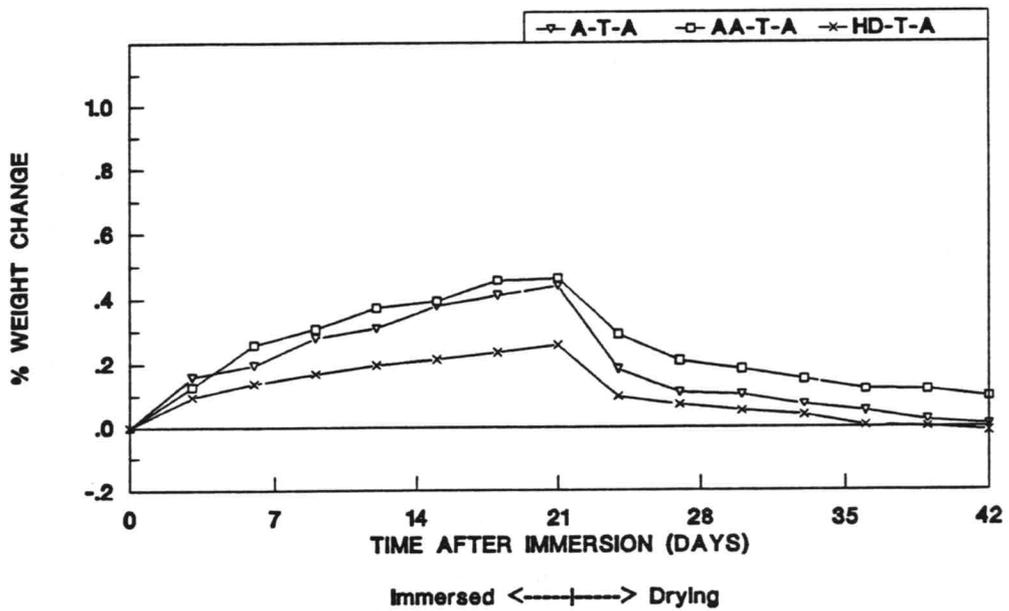


Fig. 4.7 Weight Gain/Loss of Treated Cubes (Air dried)

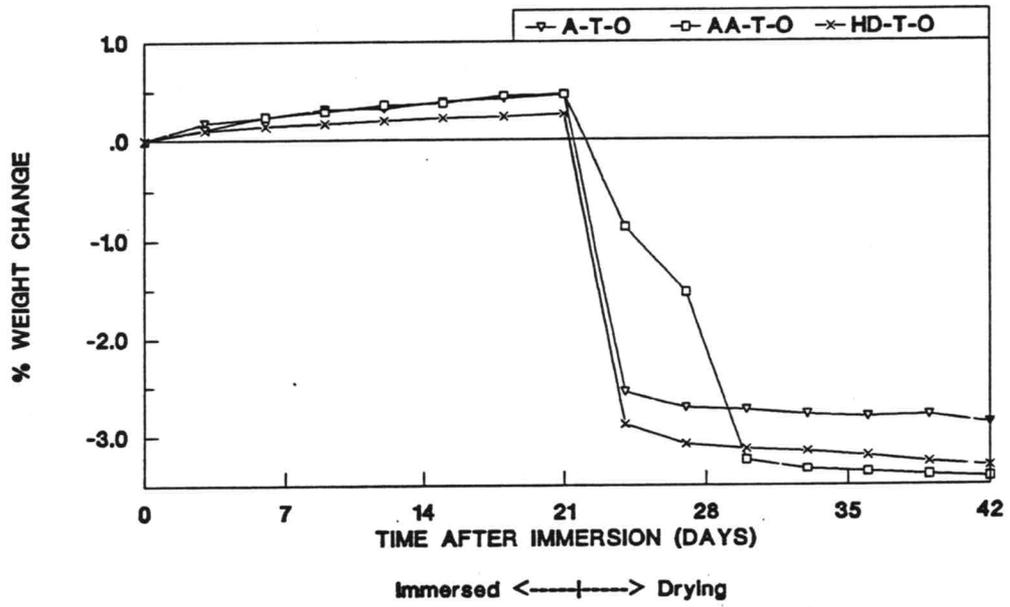


Fig. 4.10 Weight Gain/Loss of Treated Cubes (Oven Dried)

For example, mix types AA and HD exhibited the largest negative weight changes upon oven drying due to their higher moisture contents at time of immersion.

Results of the oven dried specimens are of limited use. The original intent for performing oven drying was to determine whether this procedure could be used to shorten the overall time required to perform the NCHRP test series. Conceivably, a procedure could be developed whereby a given cube could be oven dried at a specified temperature for a fixed time period. Comparison of cubes dried in the same way might provide an indication of the vapor permeability of the treated specimens. A practical limitation is that the drying process would have to be carefully controlled for each individual cube, requiring large amounts of oven space to prevent interaction of the drying rates of the various specimens. Oven drying for a sufficient time period will always result in a complete drying of the specimens, and it can be argued that this method is somewhat unrealistic, since concrete in the field is unlikely to be subjected to such elevated temperature.

4.5 CHLORIDE ION INTRUSION

Chloride absorption data were obtained from the salt ponding tests (AASHTO T259/T260) and from the NCHRP 244 test cubes. Chloride sampling for the NCHRP 244 cubes was performed by drilling with a rotary hammer. In the original NCHRP 244 study, the cubes were split and one half was crushed to a fineness sufficient to obtain chloride samples. For the present study, this procedure was found to be time consuming and the possibility of contamination relatively high due to the nature of the process. Also, the process of reducing the sample may lead to a higher crushed mortar content in the sample than in the original cube, since the aggregate does not pulverize as easily as the mortar.

A pilot study was conducted to assess the difference in chloride content in samples obtained by drilling and samples obtained by crushing. A cube was split; one half was crushed, and samples were obtained by drilling from the second half. Chloride content was found to be approximately twice as high in crushed samples than in samples obtained by drilling. This may be due to the differences in sample mortar contents and the fact that the drilling procedure excludes the top 1.6 mm (1/16 in.) of the sample (which contains large amounts of chlorides). Thus it was decided that the sampling be performed as in the salt ponding test, and a minimum of six drilled samples (2 depths for each of 3 holes) were taken from each cube. After initial results were available, additional sampling was performed from AA and HD cubes to verify the results. Also, one untreated class A AASHTO test specimen had first depth chloride concentrations much higher than

other samples from companion slabs, and was retested; results from the retested samples were found to be similar to the initial results.

Absorbed chlorides were determined by deducting base chlorides, obtained from the control AASHTO test slabs designated for each mix, from the total chlorides at the same depth. Total chlorides (acid soluble) for all samples are presented in Appendix A.

Average absorbed chlorides (ppm) were computed for samples of the same mix, depth, and treatment conditions. Averages for the salt ponding tests are shown in Fig. 4.11, and for the NCHRP test in Fig 4.12. Due to the variability in the data, conclusions based on simple comparison of the averages may not be valid. To develop more reliable conclusions, two-way hypothesis tests using the t-statistic were conducted to determine if differences between means (averages) were significant at the 5% level. It should be noted that the number of data points for each condition was relatively small (9 to 18). Also, moisture contents of specimens at treatment and at the time of exposure to salt water are uncontrolled variables which may have an effect on performance.

A summary of the statistical comparisons is presented in Table 4.4. Standard deviations and averages of all tested specimens are presented in Table 4.5. Also, 95% confidence intervals for the means (computed using \pm two standard deviations) are presented in Figs. 4.13 and 4.14 for AASHTO and NCHRP data, respectively. The large variability of the data can be seen in almost all cases; however, samples from treated specimens and from the second depth (13 mm to 25 mm) had the least variability. This may indicate that the variability was not caused by testing/sampling errors. A factor which may have contributed to the large variability is the nature of the sampling process, that is, obtaining three or more samples per depth from a relatively small number of specimens. Thus, micro level sampling was used to draw macro level conclusions. This process, combined with the inherent nonhomogeneity of concrete increased the variability. The negative values in Figs. 4.13 and 4.14 result from using the absorbed chlorides (total chlorides minus chlorides in control specimens), which was in some cases zero.

Considering differences between mixes (treated and untreated) for the AASHTO salt ponding tests (Fig. 4.11, Table 4.4), at the first depth the class HD mix absorbed the least chlorides, and mix classes A and AA appear similar. These observations were verified statistically, except no difference was observed between mix classes HD and A. An unexplained larger variation in the data occurred for mix class A, which contributed to this statistical observation. No statistical difference was observed between treated or untreated mixes at the second depth, except for untreated mix class AA vs. HD.

Comparison between treated and untreated slabs (for a given mix) indicated the difference for classes AA and HD was significant at the first depth. The absence of difference due to

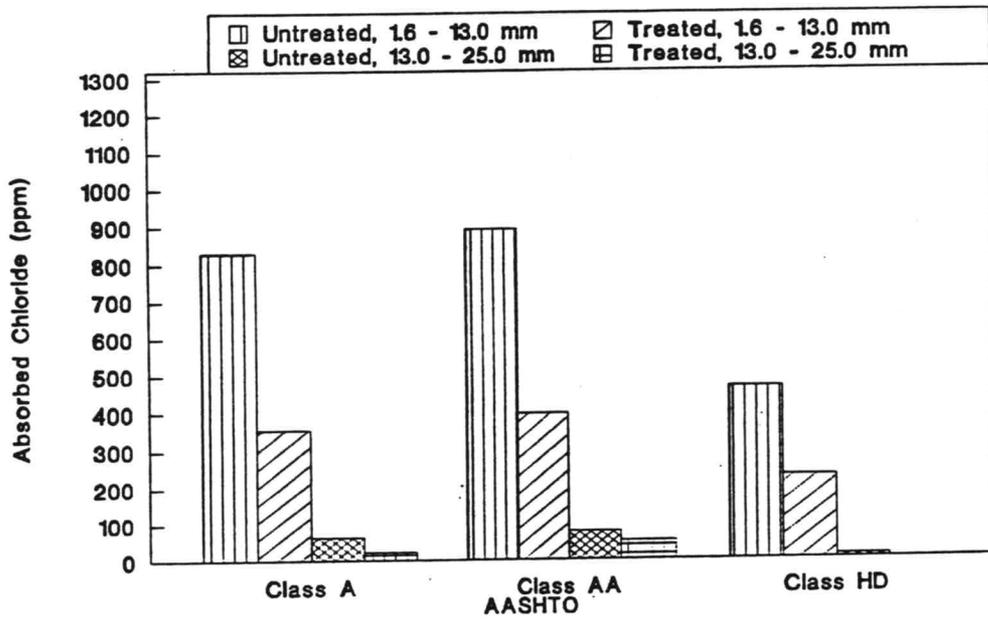


Fig. 4.11 Average Absorbed Chlorides, AASHTO T259/T260
(1 lb/cyd = 255 ppm = 0.594 kg/m³)

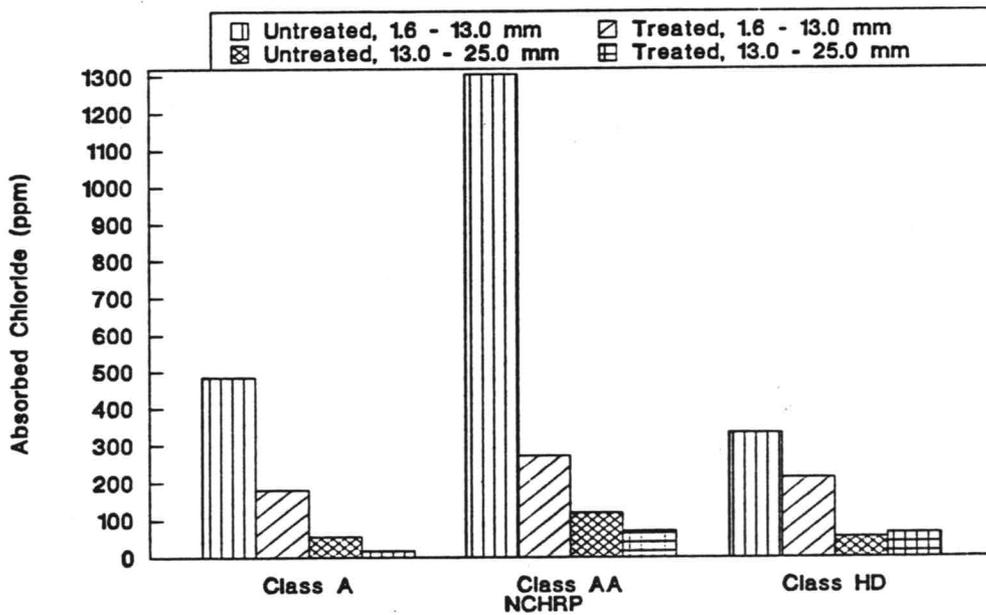


Fig. 4.12 Average Absorbed Chlorides, NCHRP 244
(1 lb/cyd = 255 ppm = 0.594 kg/m³)

Table 4.4 Samples for Which Statistical Comparison Indicated Difference Between Means

| Comparison Between | AASHTO | NCHRP |
|--|-------------|-----------------------|
| Mix Type, 1 st Depth, Untreated | AA vs. HD | AA vs. A AA vs. HD |
| Mix Type, 1 st Depth, Treated | AA vs. HD | All Similar |
| Mix Type, 2 nd Depth Untreated | AA vs. HD | All Similar |
| Mix Type, 2 nd Depth, Treated | All Similar | All Similar |
| Treatment Condition (T vs. U same mix), 1 st Depth | AA and HD | AA |
| Treatment Condition, (T vs. U same mix), 2 nd Depth | All Similar | All Similar |

T: treated, U: untreated

Table 4.5 Means and Standard Deviations for Absorbed Chlorides (ppm)

| Sample | AASHTO Test (ppm) | | NCHRP Test (ppm) | |
|---------|-------------------|-----------|------------------|-----------|
| | Mean | Std. Dev. | Mean | Std. Dev. |
| A-T-D1 | 355 | 297 | 181 | 199 |
| A-U-D1 | 829 | 932 | 485 | 498 |
| A-T-D2 | 23 | 33 | 19 | 27 |
| A-U-D2 | 64 | 118 | 57 | 150 |
| AA-T-D1 | 396 | 246 | 273 | 185 |
| AA-U-D1 | 891 | 492 | 1307 | 593 |
| AA-T-D2 | 50 | 50 | 72 | 75 |
| AA-U-D2 | 79 | 56 | 120 | 135 |
| HD-T-D1 | 228 | 109 | 214 | 206 |
| HD-U-D1 | 465 | 241 | 335 | 238 |
| HD-T-D2 | 1 | 3 | 65 | 113 |
| HD-U-D2 | 11 | 17 | 54 | 100 |

T: treated, U: untreated, D1: first depth, D2: second depth
 (1 lb/cyd = 255 ppm = 0.594 kg/m³)

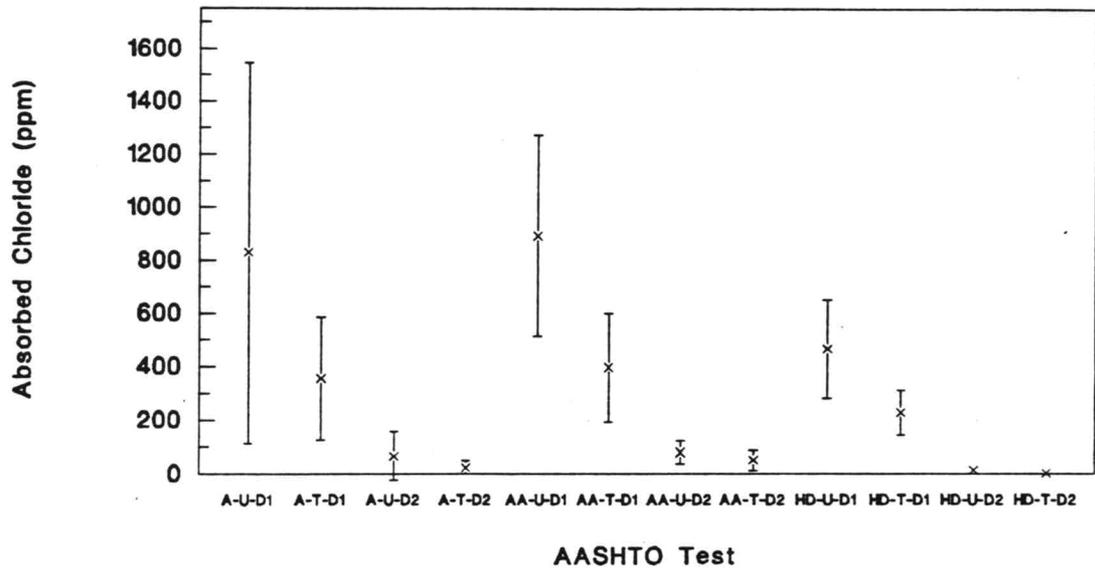


Fig. 4.13 95% Confidence Intervals for Means, AASHTO Test
 T: treated, U: untreated, D1: first depth, D2: second depth
 (1 lb/cyd = 255 ppm = 0.594 kg/m³)

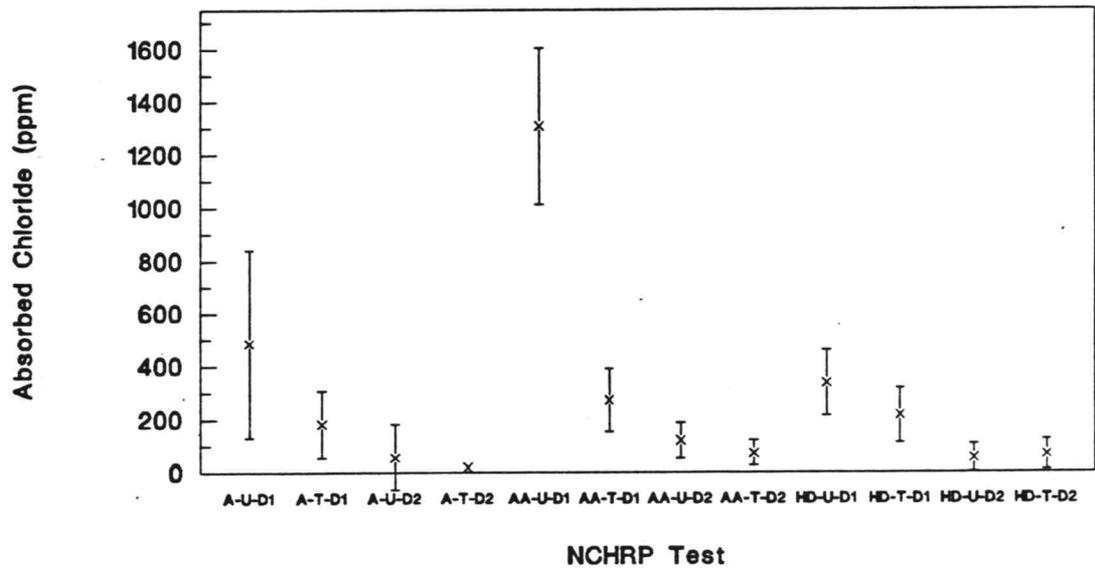


Fig. 4.14 95% Confidence Intervals for Means, NCHRP Test
 T: treated, U: untreated, D1: first depth, D2: second depth
 (1 lb/cyd = 255 ppm = 0.594 kg/m³)

treatment for mix type A is again explained by the large variability in the data. No statistical difference was observed between treated and untreated concrete, regardless of mix, at the second depth.

Examining results from the NCHRP cubes (Fig. 4.12, Table 4.4), the untreated mix class AA absorbed considerably more chlorides at the first depth than classes A and HD, which were statistically similar. No statistical difference was found between treated mixes at the first depth, or between treated or untreated mixes at the second depth. Considering treated vs. untreated cubes for a given mix, the only statistical difference observed was for mix class AA at the first depth.

Comparing results obtained from the AASHTO slabs to those of the NCHRP cubes yielded no statistically significant differences in absorbed chlorides for samples of the same mix, depth, and treatment. However, for both test procedures, the difference between chlorides measured at the first and second depths was significant for all mixes for a given treatment condition. The small measured chlorides at the second depth, regardless of treatment or mix, suggests that chloride absorption in the upper 13 mm (1/2 in.) may be a more useful indicator of performance using these tests. The observed variability in the chloride data also suggests that the cost of testing and importance of the results should be closely examined in selecting the number of specimens to be sampled.

While statistical conclusions were limited due to the variability in the data, in all but one case (NCHRP test, HD mix, second depth), mean chlorides absorbed by treated mixes were lower than for corresponding untreated mixes. This trend was consistent and should not be ignored. The fact that treatment lowered chloride absorption is clear, despite statistical conclusions, although the degree of improvement is subject to debate. Similarly, the lack of statistical difference between treated and untreated mixes at the second depth should not be interpreted to mean that silanes do not reduce chlorides at depths below 13 mm (1/2 in.). Rather, it may indicate that the tests performed do not produce sufficient chloride levels at this depth to adequately assess the improvement due to treatment.

CHAPTER 5

RESULTS OF PHASE II - SURFACE FINISH

5.1 INTRODUCTION

The performance of tine finished class AA and class HD concretes was evaluated in this portion of the study. Tests were conducted using the ODOT Series tests. Effects of the surface finish on performance were evaluated through comparison to the results from Phase I, whose specimens contained smooth or broom finishes. All results presented are averages from individual test specimens. Test results are presented according to the performance parameter measured.

5.2 DEPTH OF PENETRATION

The standard silane application rate, based on nominal surface area, was used for treatment of the specimens. The application rate was not adjusted for the increased actual surface area produced by the tine finish. Penetration followed the contours of the tine finish, so measurements of depth of penetration were made perpendicular to the surface at any given point. Measured depth of penetration for tine and broom finished specimens are presented in Table 5.1. Penetration was comparable for both finishes.

5.3 WATER ABSORPTION

Absorption results for both tine and broom finished specimens are presented in Table 5.2. As observed in Phase I, class HD concrete absorbed less water than class AA. In general, the tine finished specimens absorbed slightly more water than the broom finished specimens, although the absolute differences were not great. The effective area of the tine finished surface is larger than for a broom finished surface, resulting in a larger surface area through which absorption occurs. For the same reason, the effective silane application rate was also slightly lower for the tined specimens. Examining the percent improvement due to treatment, the difference between tine and smooth finishes was small after 48 hr, but became significant at 50 days.

Table 5.1 Depth of Penetration, Broom and Tine Finishes (mm)

| Mix Type | Tine | Broom |
|----------|------|-------|
| AA | 11 | 9.4 |
| HD | 10 | 13.5 |

(1 mm = 0.039 in.)

Table 5.2 Absorption of Broom and Tine Finished Specimens

| Mix Type | 48 hr. Untreated (%) | 48 hr. Treated (%) | Improvement (%) | 50 day Untreated (%) | 50 day Treated (%) | Improvement (%) |
|----------|----------------------|--------------------|-----------------|----------------------|--------------------|-----------------|
| AA Broom | 4.12 | 0.17 | 96 | 5.11 | 0.79 | 85 |
| AA Tine | 4.47 | 0.41 | 91 | 5.28 | 1.88 | 64 |
| HD Broom | 3.98 | 0.36 | 91 | 4.68 | 0.77 | 84 |
| HD Tine | 2.75 | 0.39 | 86 | 4.27 | 1.34 | 68 |

5.4 CHLORIDE ION INTRUSION

Averages of absorbed chlorides for the tine finished specimens are presented in Fig. 5.1. Means and standard deviations of absorbed chlorides for both tine finished and smooth finished specimens (repeated from Phase I) are contained in Table 5.3. Also, 95% confidence intervals for the means of the tine finish specimens are presented in Fig. 5.2. Statistical comparisons of the difference between means at the 5% level were also performed as in Phase I.

As compared to the smooth finished specimens of Phase I, average absorbed chlorides were generally larger for the tine finished specimens. Statistical comparisons indicated chlorides absorbed by the untreated specimens were significantly different than those of the smooth specimens, for both mixes at the first depth. However, there was no statistical difference due to surface finish for

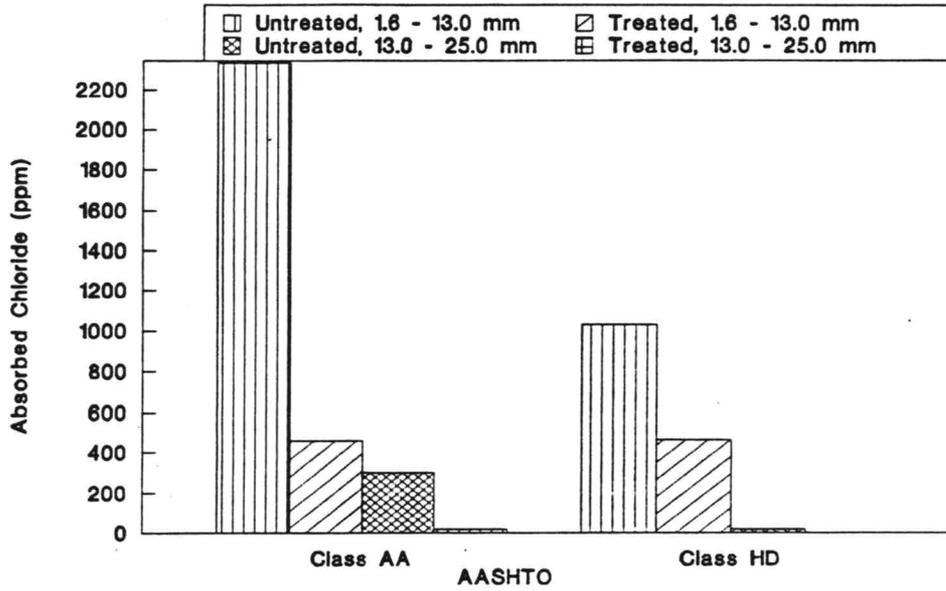


Fig. 5.1 Average Absorbed Chlorides, Tine Finish (1 lb/cyd = 255 ppm = 0.594 kg/m³)

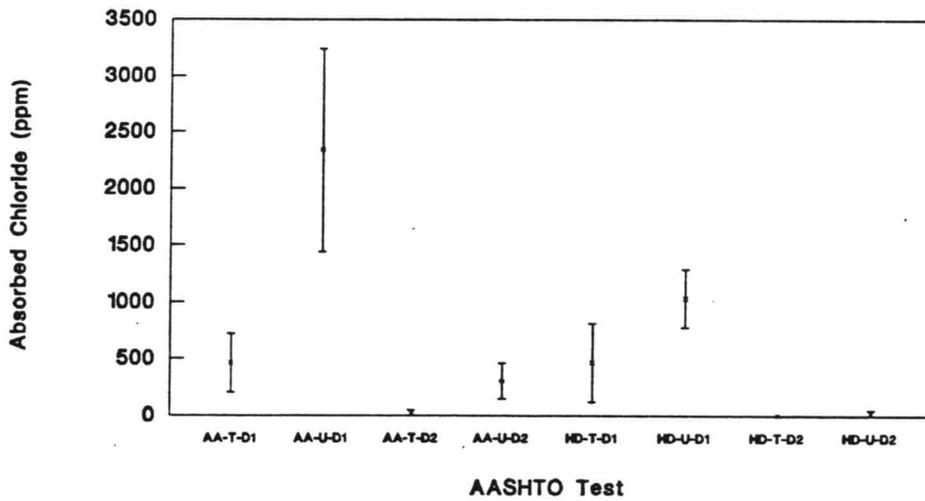


Fig. 5.2 95% Confidence Intervals for Means, Tine Finish
 T: treated, U: untreated, D1: first depth, D2: second depth
 (1 lb/cyd = 255 ppm = 0.594 kg/m³)

Table 5.3 Means and Standard Deviations for Absorbed Chlorides, Smooth and Tine Finishes (ppm)

| Sample | Mean (Smooth) | Std. Dev. (Smooth) | Mean (Tine) | Std. Dev. (Tine) |
|---------|---------------|--------------------|-------------|------------------|
| AA-T-D1 | 396 | 246 | 461 | 337 |
| AA-U-D1 | 891 | 492 | 2341 | 1173 |
| AA-T-D2 | 50 | 50 | 20 | 33 |
| AA-U-D2 | 79 | 56 | 303 | 201 |
| HD-T-D1 | 228 | 109 | 465 | 453 |
| HD-U-D1 | 465 | 241 | 1034 | 334 |
| HD-T-D2 | 1 | 3 | 0 | 0 |
| HD-U-D2 | 11 | 17 | 22 | 34 |

T: treated, U: untreated, D1: first depth, D2: second depth
 (1 lb/cyd = 255 ppm = 0.594 kg/m³)

treated specimens at the first depth. At the second depth, the only statistically significant difference due to surface finish was for untreated class AA.

Considering averages for only tine finished specimens (Fig. 5.1), the untreated class AA specimens absorbed more chlorides than class HD, while chloride levels for treated concrete appear to be similar. These observations were confirmed statistically. The difference between treated and untreated concrete was also statistically significant for both mixes at the first depth, and for class AA at the second depth.

For this study, the extreme top surface of the specimen was chosen as a reference point for measuring depths to obtain powder samples, rather than the bottom of the grooves created by the rake. This choice is justified since for a bridge deck, the path from the exposed concrete surface to the reinforcement would similarly be reduced by the depth of the tines. Thus the generally higher chloride contents for the tine finished specimens are somewhat expected, especially for the first sampling depth. A larger portion of the powder sample would be expected to contain high chlorides due to the larger surface area directly exposed to salt solution. Also, use of the top surface as a datum from drilling did not allow complete removal of surface substrate with very high chlorides since the depth of discarded sample material (0-1.6 mm (0-1/16 in.)) did not extend to the bottom of the tines.

CHAPTER 6
RESULTS OF PHASE III - CURING AND CLEANING METHODS

6.1 INTRODUCTION

The effects of curing method and cleaning prior to treatment on the performance of silane treated concrete were evaluated in this phase of the study. Three curing methods (linseed oil emulsion, pigmented membrane, self cured) and three cleaning procedures (shot-blasting, high-pressure water washing, no cleaning) were examined, and all specimens utilized class AA concrete. Primary tests were conducted on smooth finished specimens using the NCHRP 244 test procedure. Curing compounds were applied directly to the fresh concrete surface immediately after casting, and to the remaining sides of the cubes once the forms were stripped. A pilot study was also performed to examine curing and cleaning effects on a tine finish. The pilot study utilized the ODOT test procedures. Chlorides were not examined for either test series. Within each series of tests, results are presented according to the behavioral parameter measured. All results presented are averages; results for individual cubes are contained in Appendix A.

6.2 PRIMARY TESTS

6.2.1 Depth of Penetration

At the conclusion of the NCHRP 244 test, one cube from each curing/cleaning combination was split and penetration measurements taken. Penetration of the curing compounds themselves was evaluated by examining the uncleaned cubes. The linseed oil emulsion penetrated about 5 mm into the top surface of the cube, where the compound had been applied directly to fresh concrete. On the remaining cube sides (linseed applied after forms stripped), penetration of linseed oil was negligible. The pigmented membrane curing compound did not penetrate through any of the surfaces, but formed a differentiable surface layer on all cube faces.

Results for depth of penetration of silane are shown in Table 6.1. The relatively smaller recorded values (as compared to Phase I ODOT Series) are due to the fact that silane was applied to cubes with moisture contents in the range of 3%. Silane penetration into the self-cured specimens was slightly larger than for the specimens treated with a curing compound. The linseed oil cured specimens had similar penetration regardless of cleaning method, or whether cleaning was performed. The cleaned specimens cured with pigmented membrane compound performed

similarly, but penetration was reduced considerably for the uncleaned cube. The 2.2 mm silane penetration reported in Table 6.1 was measured from the faces on which the compound was applied after the forms were stripped. For the top surface of the cube (compound applied to fresh concrete), silane penetration was negligible.

Table 6.1 Depth of Penetration Results, Primary Tests

| Curing Method | Penetration (mm) | | |
|----------------------|------------------|------------|-----------|
| | Shot-Blast | Water Wash | Uncleaned |
| Linseed Oil Emulsion | 4.8 | 5.2 | 5.5 |
| Pigmented Membrane | 4.9 | 5.0 | 2.2 |
| Self | 6.3 | 5.6 | - |

(1 mm = 0.039 in.)

The results indicate that it is imperative to adequately clean the surface of concrete cured with the pigmented curing compound prior to silane treatment in order to achieve penetration. Linseed oil, however, did not adversely affect penetration if the compound was applied to fresh concrete. It is important to note the cubes were not subject to UV radiation at any stage of testing. It is possible that curing of the linseed oil would take place under this condition, and the need for thorough cleaning could become more important.

6.2.2 Water Absorption

Average percent weight gained/lost upon immersion in 15% NaCl solution for treated class AA cubes is shown in Figs. 6.1, 6.2, and 6.3, for linseed, pigmented membrane, and self-cured cubes, respectively. For comparison purposes, results from Phase I for class AA concrete (self-cured, lightly sandblasted to remove surface laitance) are superimposed on each plot, and represented by the dashed curve.

Considering the cubes cured using linseed oil emulsion (Fig. 6.1), performance was similar for the three cleaning conditions, although the uncleaned cubes exhibited slightly higher absorption in early stages. As shown in Fig. 6.1, by 21 days the percent weight gained was equivalent to that of the cleaned cubes; however, this may be partially attributable to leaching of the uncleaned linseed oil from the surfaces of the cubes. An orange coloration of the immersion solution was noted, implying that measured cube weights were perhaps slightly underestimated by the weight of linseed oil lost into solution. Therefore the percent weight change curve for the uncleaned specimens may

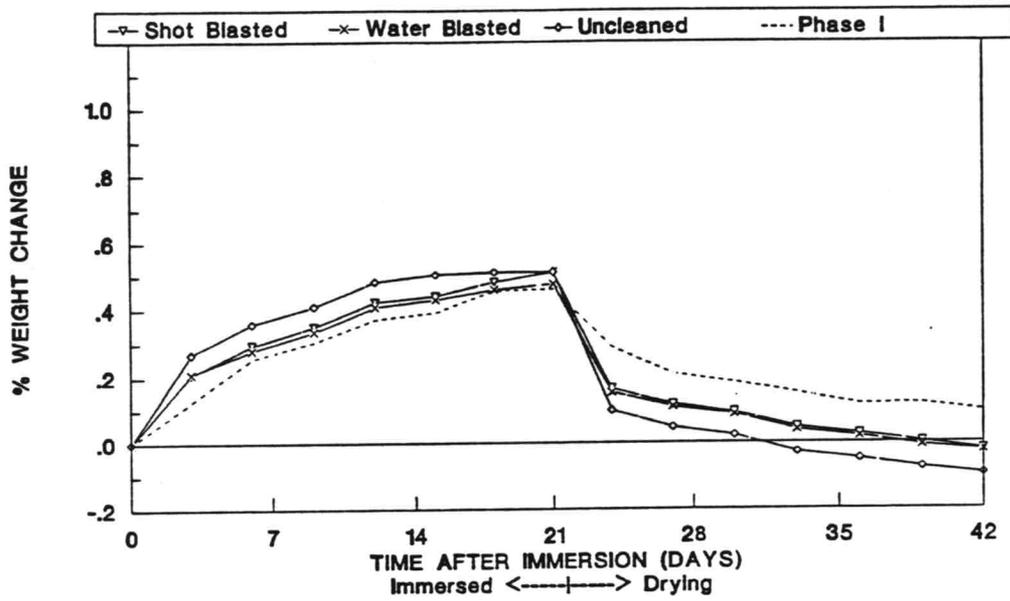


Fig. 6.1 Weight Gain/Loss of Linseed Oil Cured Cubes

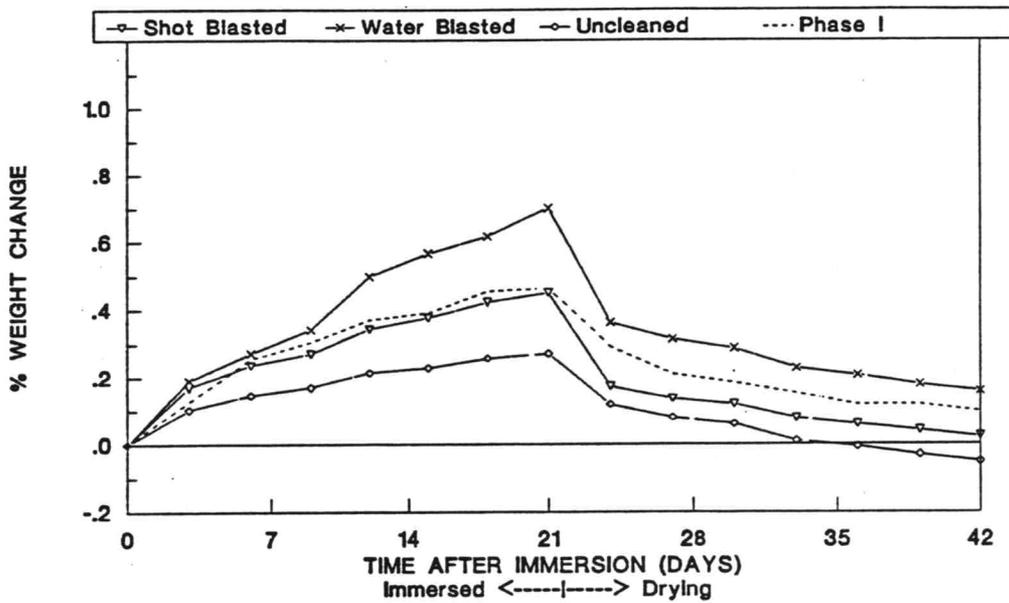


Fig. 6.2 Weight Gain/Loss of Pigmented Membrane Compound Cured Cubes

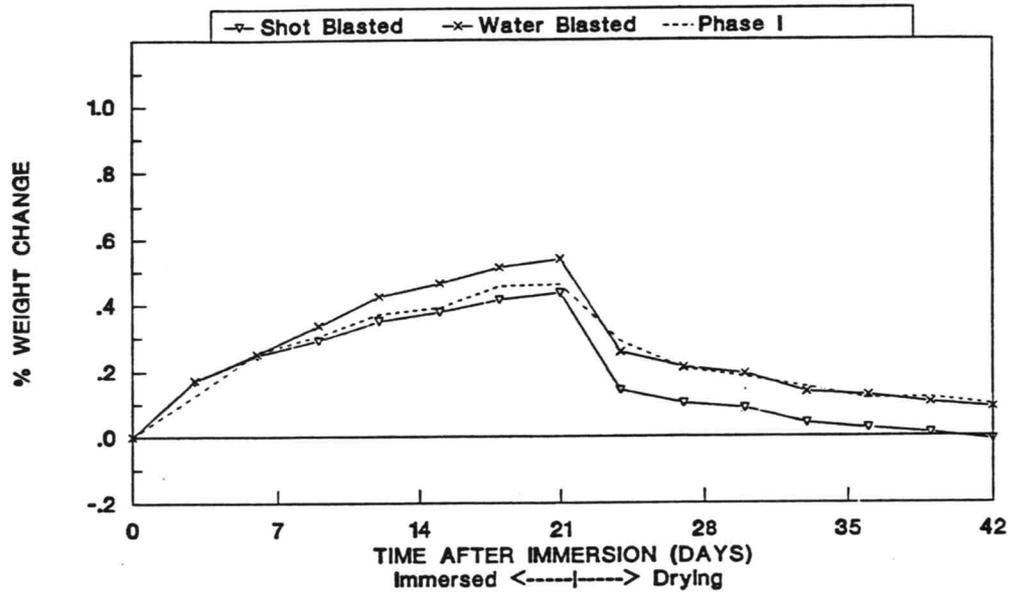


Fig. 6.3 Weight Gain/Loss of Self-Cured Cubes

be slightly higher than shown in the Figure. Absorption was found to be slightly higher for linseed oil cured specimens than for self-cured specimens of the same mix (dashed curve).

Differences in behavior were observed for the specimens cured using the pigmented membrane compound (Fig. 6.2). The curve for the high-pressure water washed specimens diverges after 10 days immersion. An unexplained increase in weight gain for one of the three cubes is responsible for the shift in the curve. The other two water-blasted cubes exhibited behavior much closer to that of the specimens cleaned by shot-blasting. Excluding the divergent specimen, the water-blasted and shot-blasted cubes performed similar to each other, and to the Phase I results. The uncleaned specimens exhibited considerably lower weight gain than the cleaned specimens. This behavior is likely attributed to the water screening ability of the membrane curing compound, either alone or in combination with the silane.

Self-cured specimens subjected to the two cleaning methods were tested to isolate behavior caused by the cleaning itself (Fig. 6.3). Water blasted specimens absorbed slightly more water than shot-blasted specimens, although the difference was not great.

6.2.3 Moisture Vapor Permeability

Examination of the air drying branches of the curves in Fig. 6.1-6.3 provides an indication of the vapor transmission characteristics of the specimens. With the exception of the divergent curve in Fig. 6.2 (explained previously), percent weights lost upon air drying for the cured/cleaned specimens were comparable to the results of Phase I. It appears that the vapor permeability characteristics were largely unaffected by the curing and cleaning methods examined.

6.3 PILOT STUDY

Tine finished slabs were cured using linseed oil emulsion (cleaned and uncleaned prior to treatment) to examine the combined effect of these variables. Depth of penetration and absorption were evaluated using the ODOT Series procedures, and cleaning was performed by shot-blasting. Results of the study were also compared to those of Phase II (tine finish, wet cured) and Phase I (smooth finish, wet cured).

6.3.1 Depth of Penetration

As in the primary study, linseed oil emulsion was applied directly to the freshly cast concrete surface. Specimens underwent oven drying prior to treatment (as per the test procedure), but were not exposed to UV radiation. The depth of penetration results presented in Table 6.2

indicate that as in the primary study, the linseed oil did not inhibit the depth of penetration of silane for the tine finish. Interestingly, penetration was slightly higher for linseed oil cured specimens, and highest for the shot-blasted tine finished specimen (roughest exposed surface).

Table 6.2 Depth of Penetration, Pilot Study

| Curing, Cleaning, Surface Finish | Depth Of Penetration (mm) (type AA slabs) |
|----------------------------------|--|
| Water, uncleaned, smooth | 9.4 |
| Water, uncleaned, tine | 11.0 |
| Linseed, uncleaned, tine | 12.3 |
| Linseed, shot-blast, tine | 13.3 |

(1 mm = 0.039 in.)

6.3.2 Water Absorption

Results of absorption tests are shown with those of Phases I and II in Table 6.3. The use of the linseed oil curing compound did not significantly alter the results as compared to the tine finished, water cured specimens of Phase II. Both tine finished cases (linseed oil cured, water cured) absorbed more water than the smooth finished specimens of Phase I. Also, the uncleaned linseed cured specimen absorbed slightly more water than the cleaned specimen.

Table 6.3 Absorption Results, Pilot Study

| Curing, Surface Finish (AA mix) | 48 hr. Abs.(%) Shot-Blasted | 48 hr Abs.(%) Uncleaned | 50 day Abs.(%) Shot-Blasted | 50 day Abs.(%) Uncleaned |
|------------------------------------|-----------------------------------|-------------------------------|-----------------------------------|--------------------------------|
| Water, broom | - | 0.17 | - | 0.79 |
| Water, tine | - | 0.41 | - | 1.88 |
| Linseed, tine | 0.44 | 0.71 | 1.30 | 1.69 |

6.4 SUMMARY

Both shot-blasting and high pressure water washing appear to be equally effective in removal of the curing compounds prior to treatment. Strictly from a standpoint of silane performance, use of shot-blasting may be preferable since the concrete moisture content is unaltered by the cleaning process. Performance of cleaned specimens cured using either compound was fairly similar to self-cured specimens of Phase I. However, the differences in behavior that might be caused by exposure to UV radiation prior to cleaning were not examined.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 INTRODUCTION

The effects of field variables on the performance of concrete treated using a generic silane water repellent material were evaluated using alternate testing procedures. The field variables examined were: 1) concrete mix design, 2) concrete surface finish, and 3) curing and cleaning methods prior to silane treatment. Three concrete mixes were examined, designated class A (similar to laboratory reference concrete, w/c = 0.49), class AA (w/c = 0.44), and class HD (high density overlay, w/c = 0.33). Surface finishes examined included smooth/broom and tine. Curing methods studied included use of linseed oil emulsion, white pigmented membrane curing compound, and self-curing. Cleaning methods examined to remove the curing compounds prior to treatment were shot-blasting, high pressure water washing, and no cleaning.

Tests were conducted using alternate laboratory procedures to examine the basic performance parameters of depth of penetration, absorption, moisture vapor permeability, and chloride ion intrusion. Test procedures used included: 1) ODOT Series Tests (OHD-L40, OHD-L35, ASTM C642, AASHTO T259/T260), and 2) tests based on NCHRP 244 Series II. Results of the study are presented according to phases of the research conducted, followed by observations concerning testing procedures and recommendations for future research.

7.2 PHASE I - CONCRETE MIX DESIGN

1. Depth of penetration was good for all mixes when treatment was applied to dry (0% moisture) concrete. The highest penetration was observed for class HD (13.5 mm), followed by class A (11.9 mm), and class AA (9.4 mm). The presence of moisture at the time of treatment was shown to adversely affect penetration, with significant reductions in penetration for all mixes occurring at moisture contents of 2-3%.
2. Retreatment of specimens which initially exhibited negligible penetration (due to treatment at very high moisture contents) was found to result in satisfactory penetration. Retreatment at full coverage rate resulted in penetrations comparable to specimens initially treated at 0% moisture. These tests were conducted on specimens which were oven-dried prior to retreatment.

3. Due to considerable variability in the chloride data, statistical evaluations indicated significant improvement due to treatment for only mix classes AA and HD using the AASHTO salt ponding data. For NCHRP cube tests, only mix class AA indicated statistically significant improvement due to treatment at this depth. Statistical observations were drawn by conducting hypothesis tests on the differences between means at the 5% level ($\alpha = 0.05$). However, in essentially all cases, mean absorbed chlorides in the first 13 mm (1/2 in.) depth were reduced due to silane treatment. This general trend should be recognized, despite statistical observations. Only very small amounts of absorbed chlorides were measured at the second depth for all mixes, regardless of treatment or test procedure.
4. The treated class HD mix outperformed treated specimens of the other two mixes, exhibiting greater depth of penetration and generally lower water and chloride absorption. Improvement in performance resulting from silane treatment was usually not as great as for the other mixes; tests on untreated specimens indicated inherently better performance of the HD mix.
5. Performance of **treated** mix classes A and AA was fairly similar, with some relative behavior affected by the test procedure used to obtain the physical parameter of interest. For treated NCHRP 244 cubes, mix class A slightly outperformed mix class AA with respect to water absorption and vapor permeability, but had similar absorbed chlorides. In the ODOT test series, mix class A exhibited greater depth of penetration and similar chloride absorption, but higher water absorption (despite its greater depth of penetration, and contrary to the trend observed in the NCHRP 244 cube tests). For both test procedures, the class AA mix exhibited a larger percent reduction in water absorption due to silane treatment.
6. Performance of **untreated** mix types A and AA was similar for the ODOT test series. The NCHRP series tests indicated superior performance of untreated mix type A over mix type AA in terms of moisture gain and chloride absorption.

7.3 PHASE II - SURFACE FINISH

Tests in this phase of the research were conducted using mix classes AA and HD. In general, performance of treated tine finished specimens was only slightly diminished as compared to smooth/broom finished specimens.

1. Penetration of tine finished specimens was found to be comparable to that of the broom finished specimens of Phase I. The depth of the hydrophobic layer followed the contour of the surface finish.
2. Water absorption of treated specimens with a tine finish was slightly higher than for companion broom finished specimens, although the differences were not great. Percent improvements due to treatment of the tine finished specimens were fairly comparable to those of broom finished specimens at 48 hr immersion, but significantly reduced at 50 days.
3. Absorbed chlorides were higher, on average, for tine finished specimens than for similar smooth finished specimens. Statistical analyses indicated the differences were significant for untreated mixes, but not for treated specimens. Untreated class HD concrete absorbed lower chlorides at the first depth, but chlorides at this depth for treated class HD and class AA mixes were statistically similar. Measured chlorides at the second depth were slightly higher than in Phase I, but still small.

7.4 PHASE III - CURING/CLEANING PROCEDURES

Tests in this portion of the study were conducted using mix class AA. Curing compounds were applied directly to fresh concrete, and test specimens were not subjected to UV radiation.

1. Performance of specimens cured by linseed oil emulsion or pigmented membrane compound was comparable to that of self-cured specimens with respect to silane penetration, water absorption, and vapor permeability, when the compounds were cleaned from the surface prior to treatment.
2. For uncleaned specimens, the presence of pigmented membrane curing compound adversely affected silane penetration, but decreased absorption due to the water screening capabilities of the curing compound. The presence of linseed oil applied to **fresh concrete** did not adversely affect penetration. Possible differences in behavior due to exposure of the curing compounds to UV radiation, or due to linseed oil application at a later age, were not examined.
3. Both shot-blasting and high pressure water washing were effective in cleaning the concrete surface prior to silane treatment. Strictly from a silane performance standpoint, shot-blasting may be preferred in the field since it does not increase the moisture content of the substrate.

7.5 OBSERVATIONS CONCERNING TEST METHODS

1. Water absorption results of NCHRP 244 series cubes and ODOT series slabs are not comparable due to differences in specimen moisture content at treatment and immersion, surface area to volume ratio, duration of immersion, and immersion solution. In all cases, the percent improvement in absorption due to treatment was lower for NCHRP 244 series cubes than for ODOT Series slabs. It is not clear whether this observation was due to the lower silane penetration for the cubes, or due to one or more of the factors mentioned above.
2. For both NCHRP 244 cubes and ODOT series slabs, the percent reduction in absorption due to treatment decreased with time. This trend indicates a more conservative estimate of improvement due to treatment is obtained for longer exposure periods. It appears that the most significant effect silane has on absorption is reduction in its rate.
3. Mean levels of absorbed chlorides at the first depth for NCHRP 244 cubes were statistically similar to those of the AASHTO salt ponding slabs. It appears that the cubes' higher surface area to volume ratio and higher salt content in the immersion solution somewhat counteracted the shorter immersion period.
3. Absorbed chlorides at the second depth were usually very small for both tests, regardless of treatment. Given the large variability in the chloride data, for the test procedures and concrete mixes examined, more effective use of testing could probably be made by collecting more samples at the first depth, and omitting chloride sampling at the second depth. Agencies should carefully consider testing costs and importance of the data in choosing the number of chloride samples for analysis.
4. Of the procedures examined, the NCHRP 244 Series II cube tests more closely represented field conditions, in terms of specimen moisture content at treatment and immersion. Substrate moisture content was shown to have a significant impact on silane penetration; it is not known to what degree other performance characteristics are influenced by penetration depth. However, variability in test conditions exists since specimen moisture content is not carefully controlled by the test procedure.
5. Results of this study point to the need to duplicate field conditions in the laboratory, to the extent possible (more so for concrete mix design than the other variables examined), in order to better predict field performance of concrete treated with silane.

7.6 RECOMMENDATIONS FOR FUTURE RESEARCH

Results of the tests indicate that the interrelationship between performance characteristics of concrete treated with silane is not straightforward. Use of a relatively more "dense" mix (lower w/c ratio and higher cement factor) did not necessarily reduce penetration or absorption, and smaller depth of penetration did not necessarily lead to higher absorption. There is a need for a more thorough understanding of the mechanisms responsible for performance.

Much attention is paid to depth of penetration of silane, perhaps since it is easily measured and provides an indication of coverage and durability (wear life) of the treatment. It would be desirable to determine to what degree, if any, penetration affects water and chloride absorption. Such knowledge would aid in establishing better limits for minimum acceptable penetration.

Acceptance limits and testing procedures for moisture vapor permeability need to be further defined if quantification of this parameter is deemed essential. A possible approach may be comparison of the rate and amount of moisture loss of treated specimens to those of untreated specimens with the same absorbed moisture. This comparison should be made under temperature, humidity, and treatment conditions similar to those expected in the field.

Drilling and testing for chlorides was found to be labor intensive and expensive. Large variations were observed in the resulting data, possibly due to local variations in chloride distribution and makeup of the powder sample (presence or absence of aggregate). An alternate approach for consideration is to measure the depth of penetration of chlorides by applying a chemical dye sensitive to a specific concentration of chloride ions. However, use of this method would require prior quantification of base chloride ion concentration in order to properly select the sensitivity of the chemical dye (for measurement of chlorides absorbed).

Finally, laboratory criteria for acceptance limits must be based on observed field performance. Studies using closely coordinated field and laboratory evaluations are needed to produce the necessary data for correlation.

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APPENDIX A

TEST DATA

A.1 WEIGHT CHANGE DATA FOR NCHRP 244 TEST SPECIMENS

Test data for individual cubes are presented in plots containing the percent weight change versus time, with three cubes presented in each plot (unless combined data are presented for comparison). The legend in each plot identifies the specimens presented.

For Phase I results, three plots are presented on each page, one for each mix type (A, AA, and HD). Figures are grouped by batch and test condition. Unless otherwise noted, cubes were lightly sandblasted prior to treatment. For Phase III results, all specimens utilized class AA concrete. Plots are grouped by curing method, and each figure contains results for a particular cleaning procedure. Cubes 1-3 (T: top graph) were shot-blasted, cubes 4-6 (M: middle graph) were high pressure water washed, and cubes 7-9 (B: bottom graph) were not cleaned.

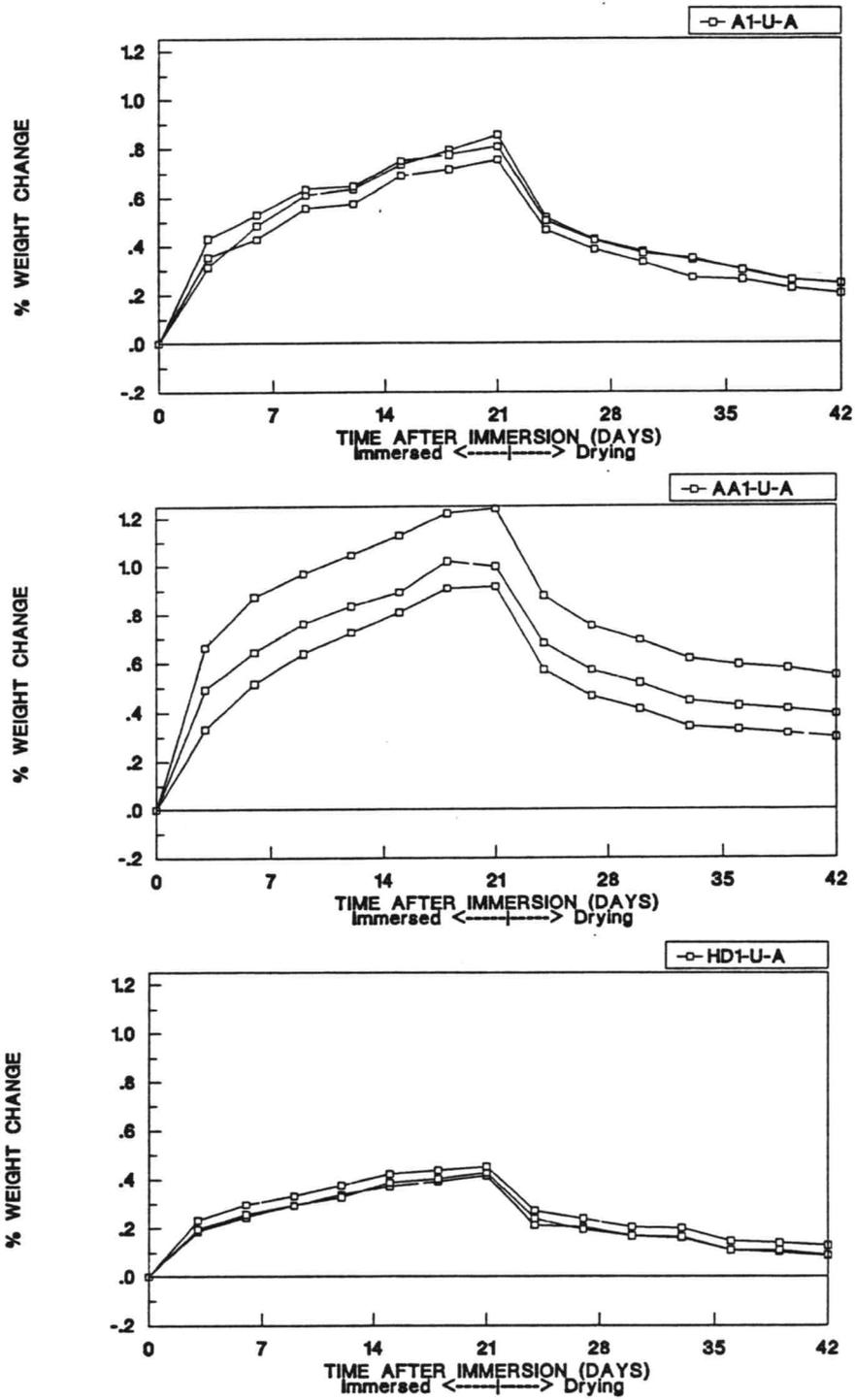


Fig. A.1 Batch #1, Untreated, Air Dried, Phase I

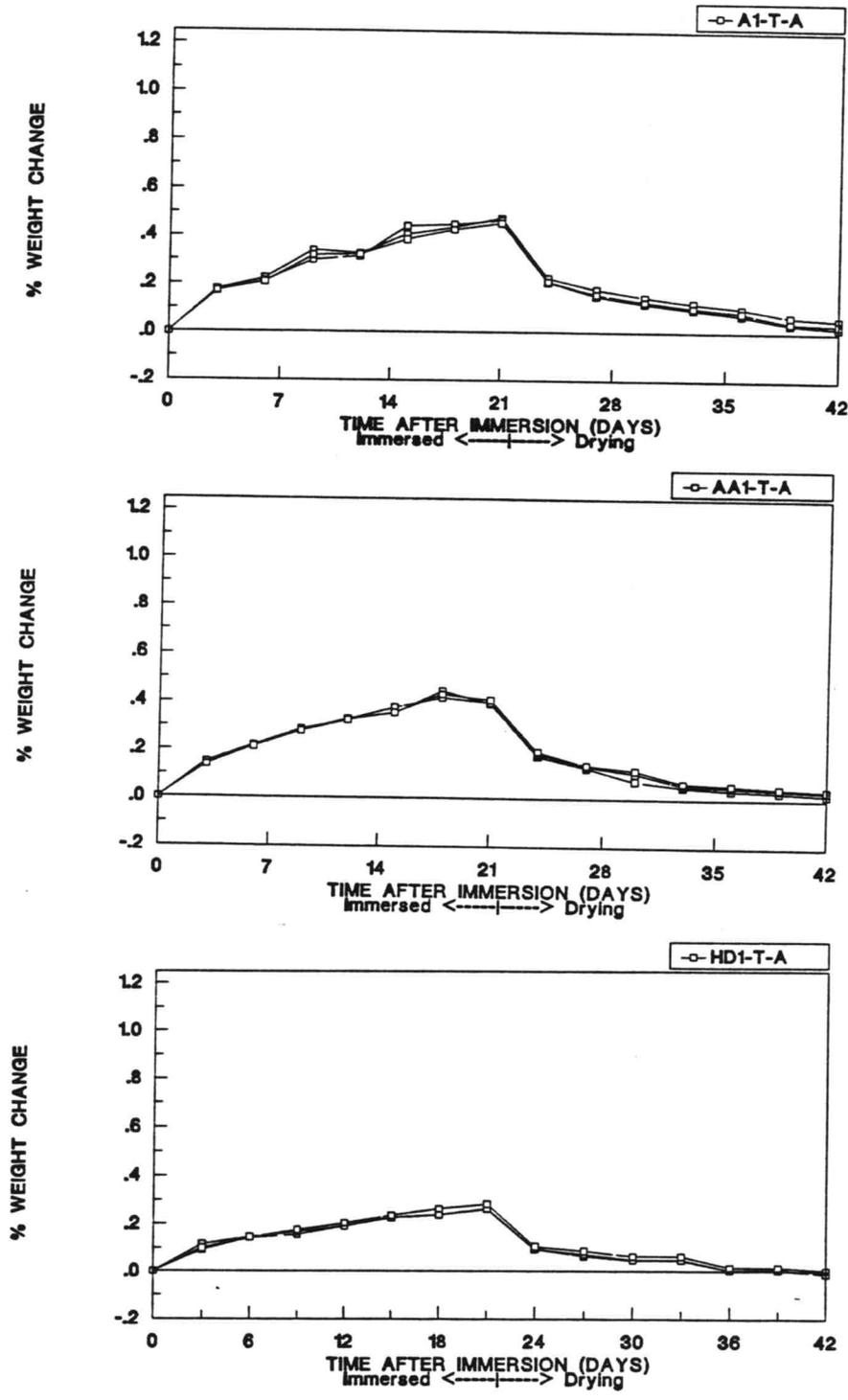


Fig. A.2 Batch #1, Treated, Air Dried, Phase I

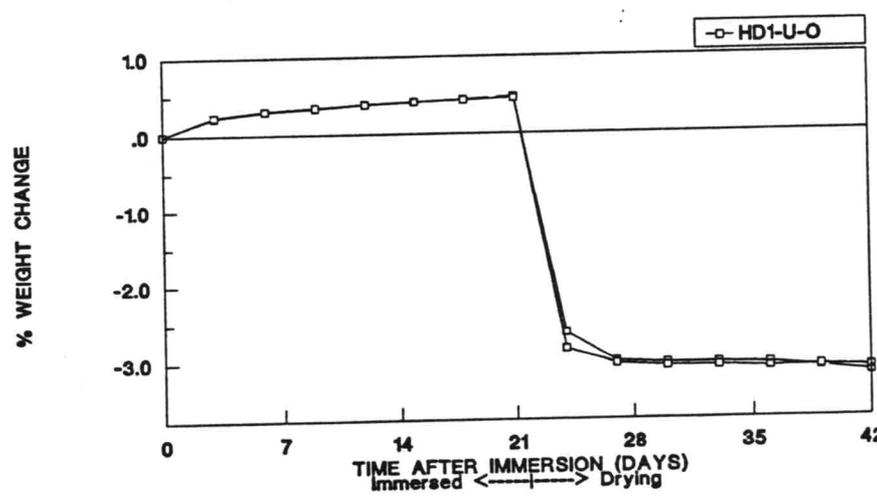
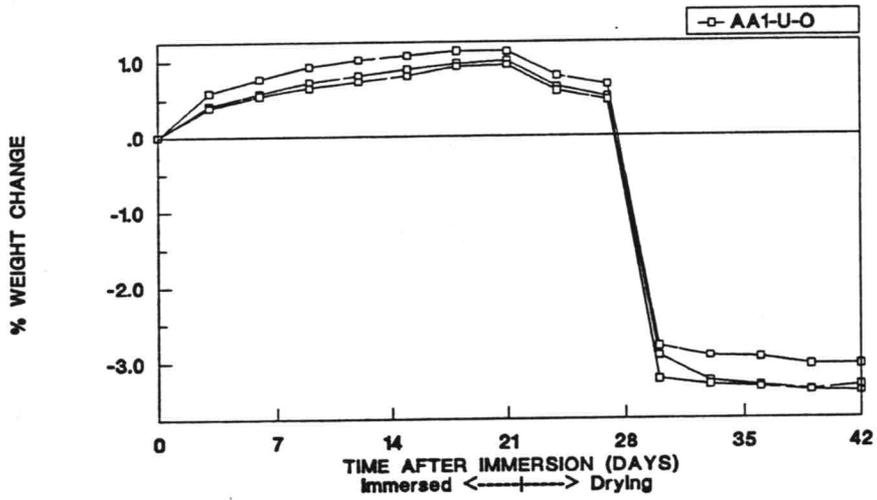
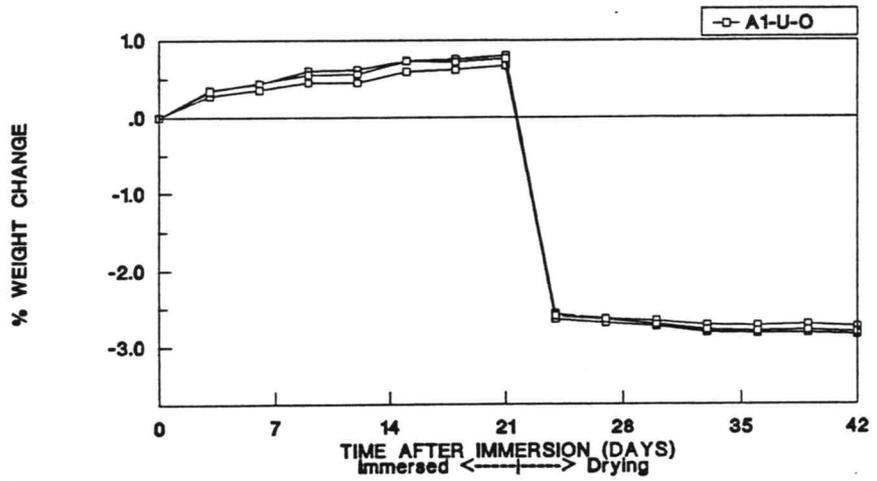


Fig. A.3 Batch #1, Untreated, Oven Dried, Phase I

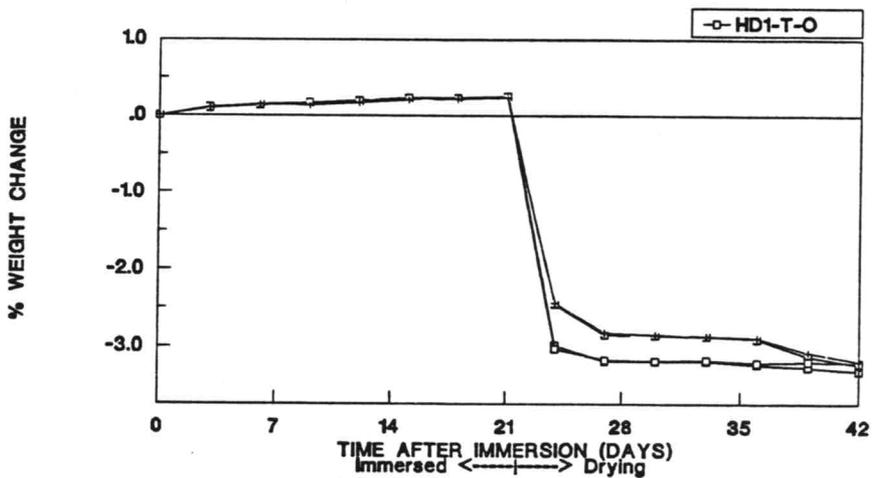
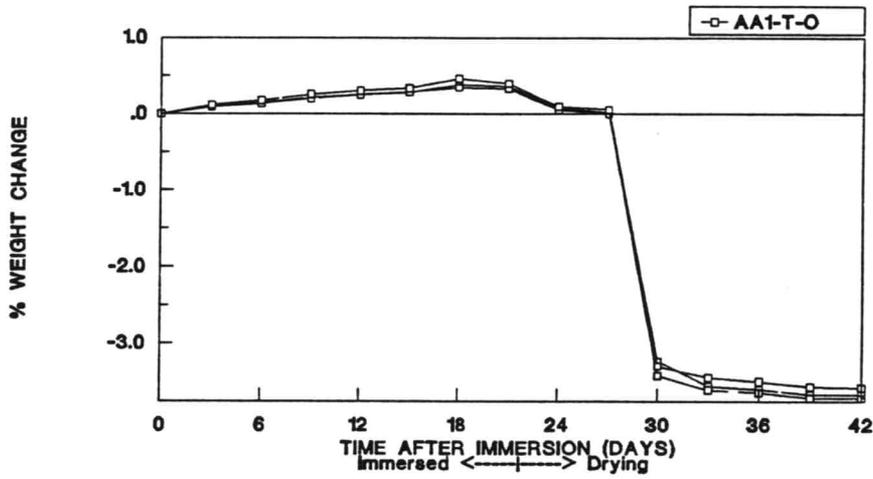
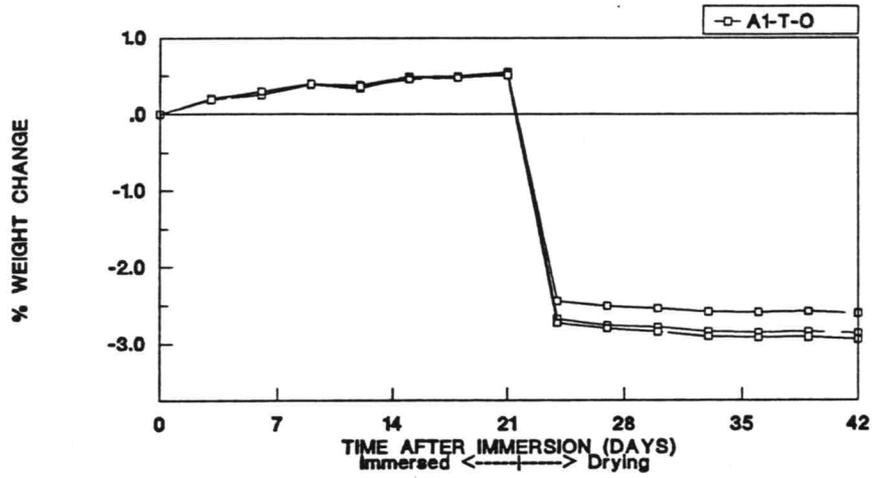


Fig. A.4 Batch #1, Treated, Oven Dried, Phase I

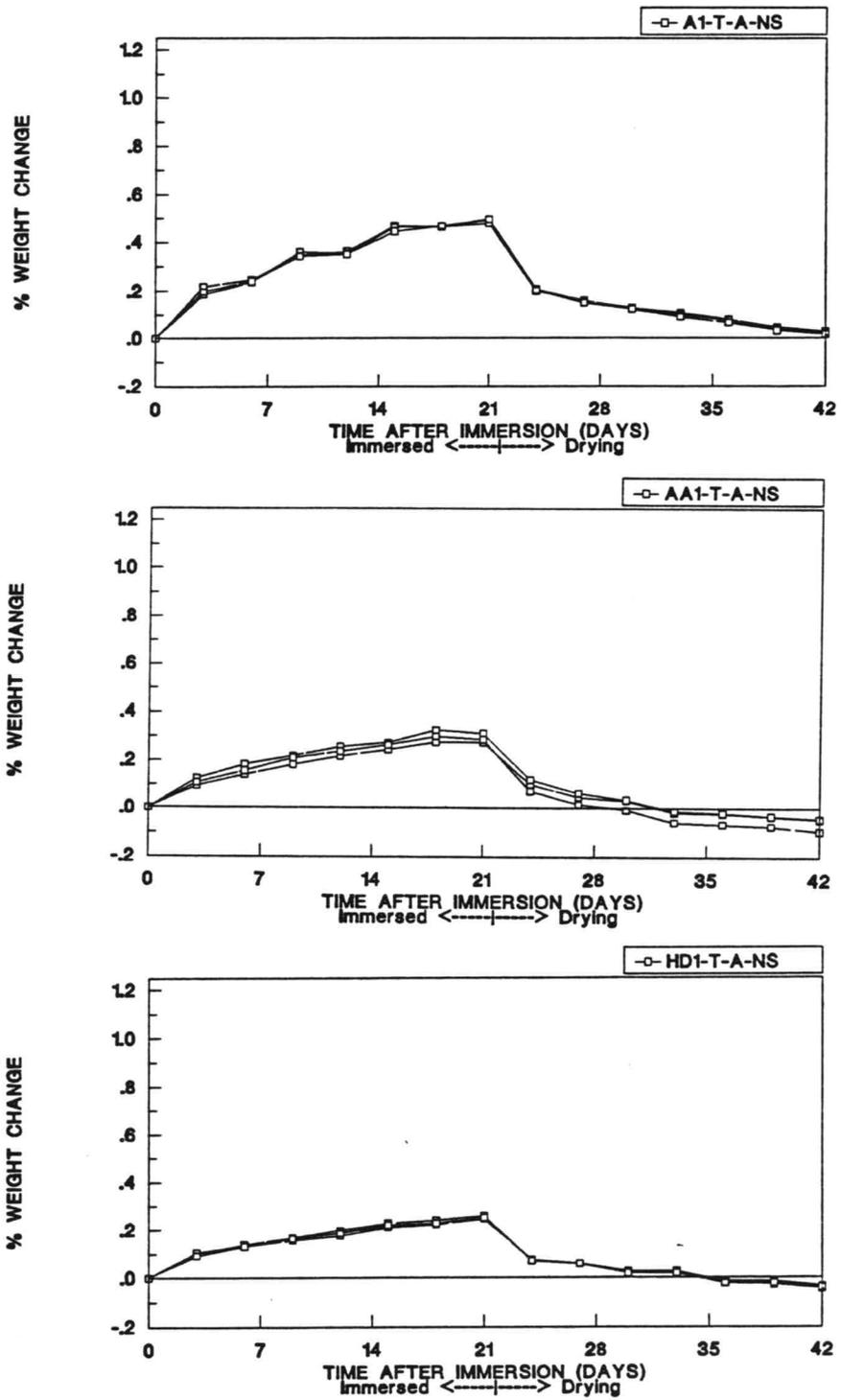


Fig. A.5 Batch #1, Treated, Air Dried, Not Sandblasted, Phase I

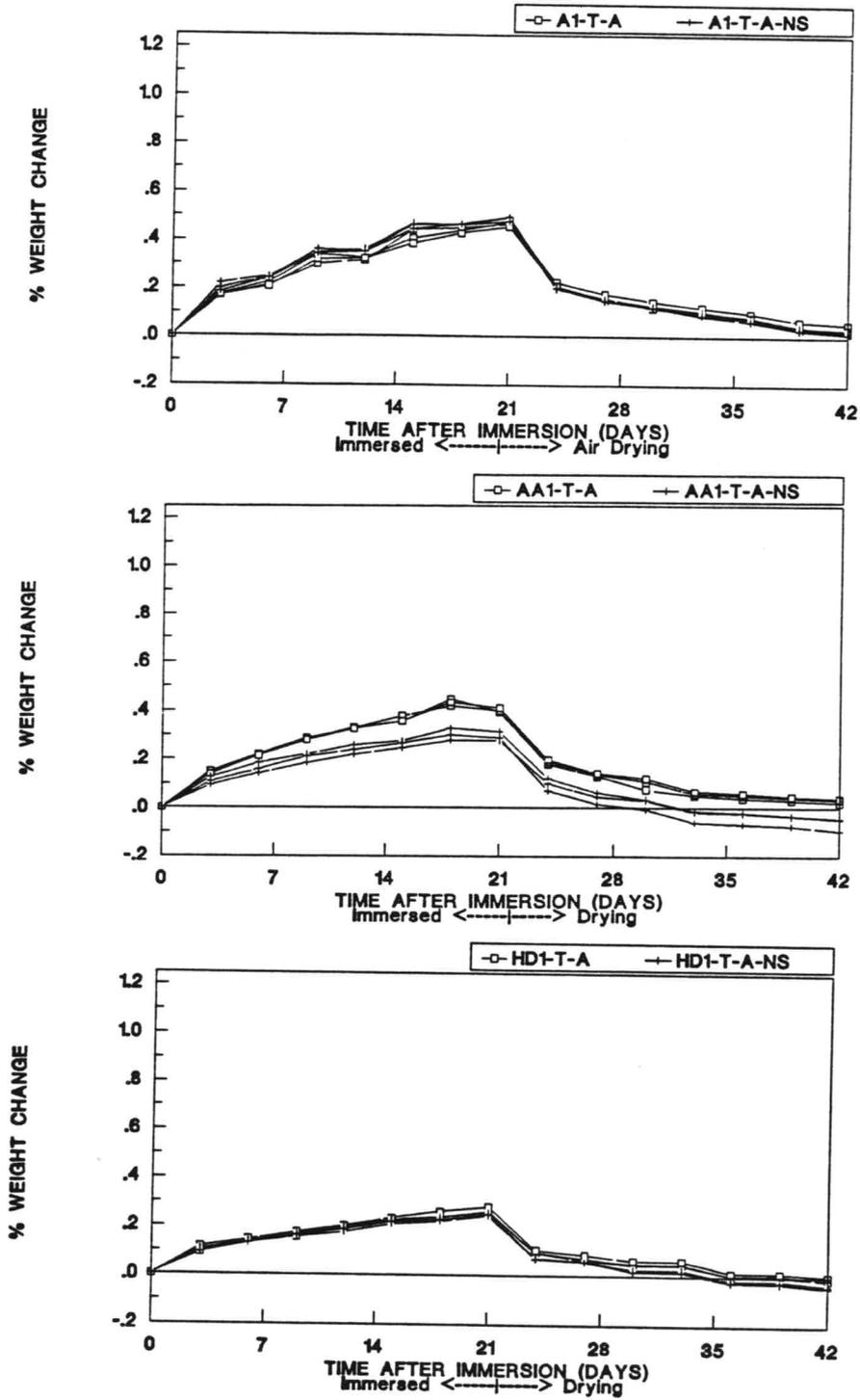


Fig. A.6 Batch #1, Treated, Air Dried, Sandblast + Not Sandblasted, Phase I

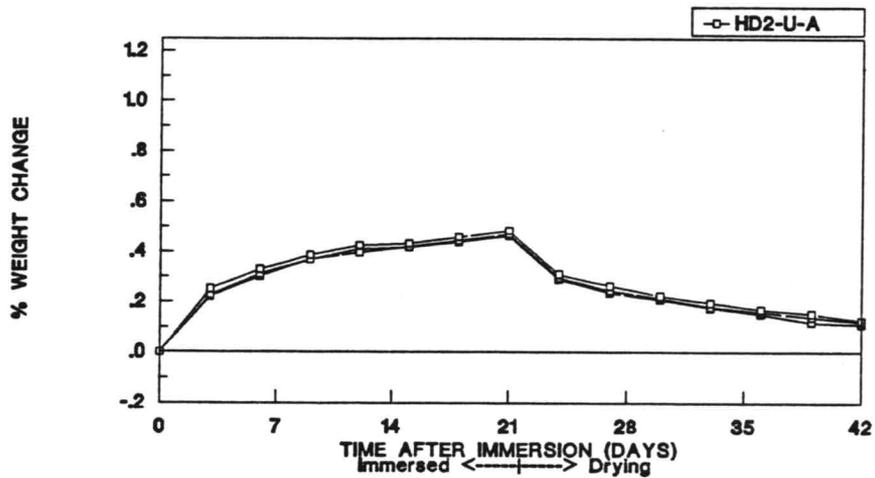
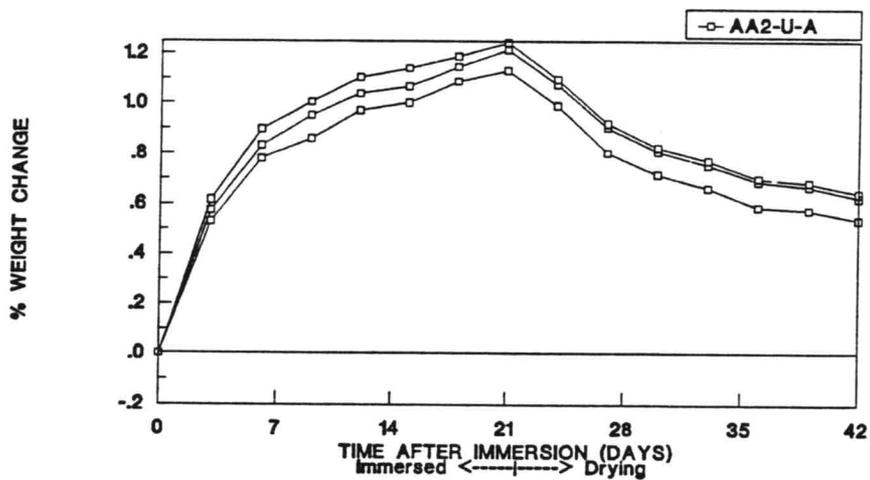
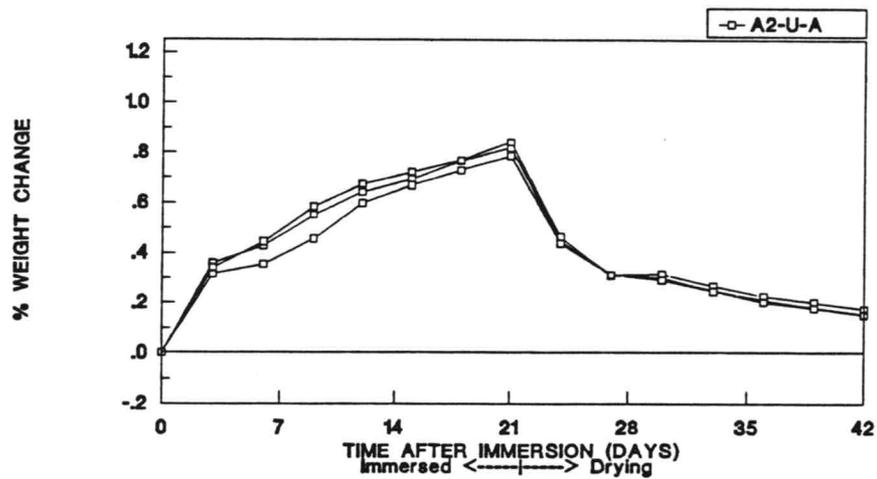


Fig. A.7 Batch #2, Untreated, Air Dried, Phase I

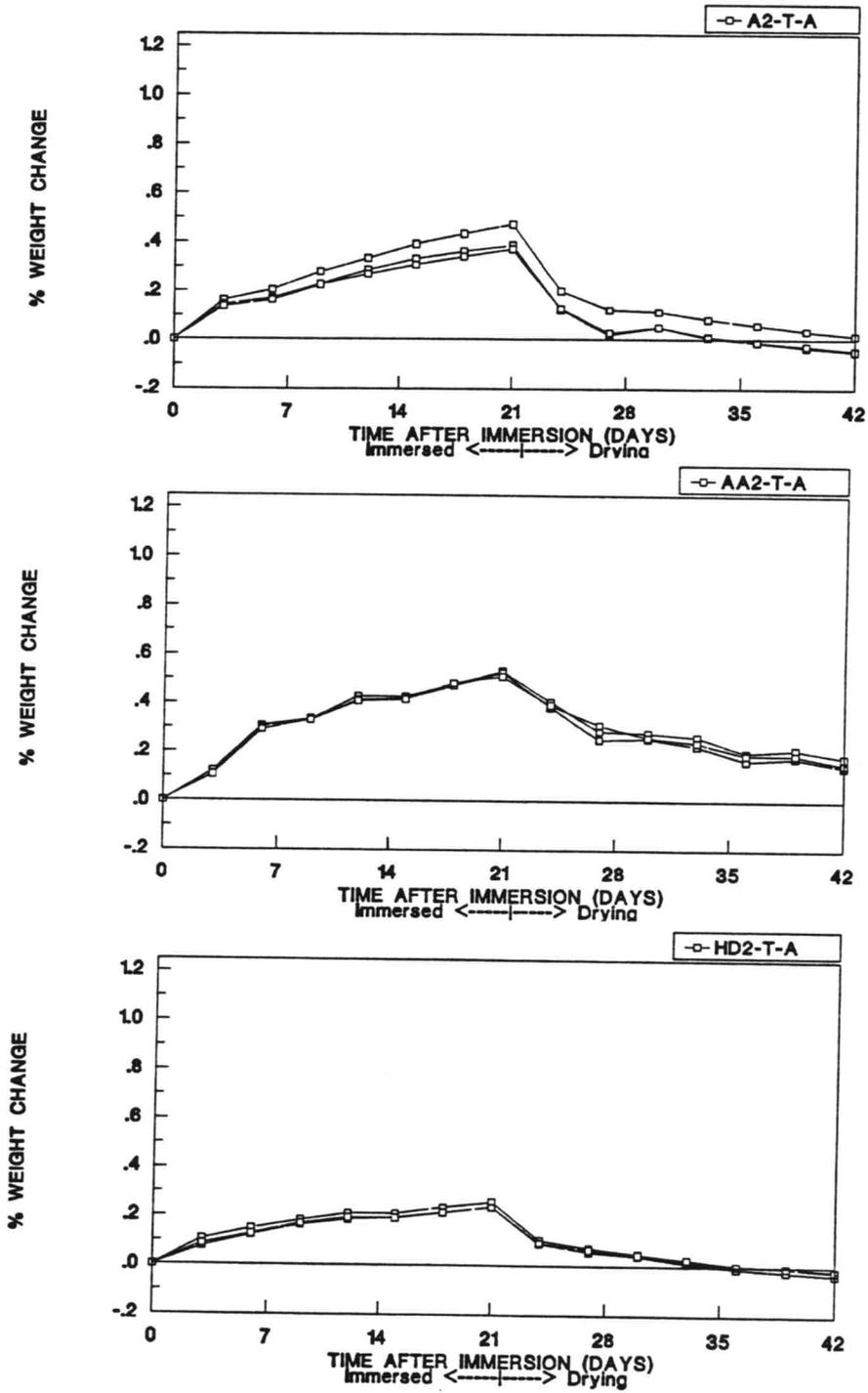


Fig. A.8 Batch #2, Treated, Air Dried, Phase I

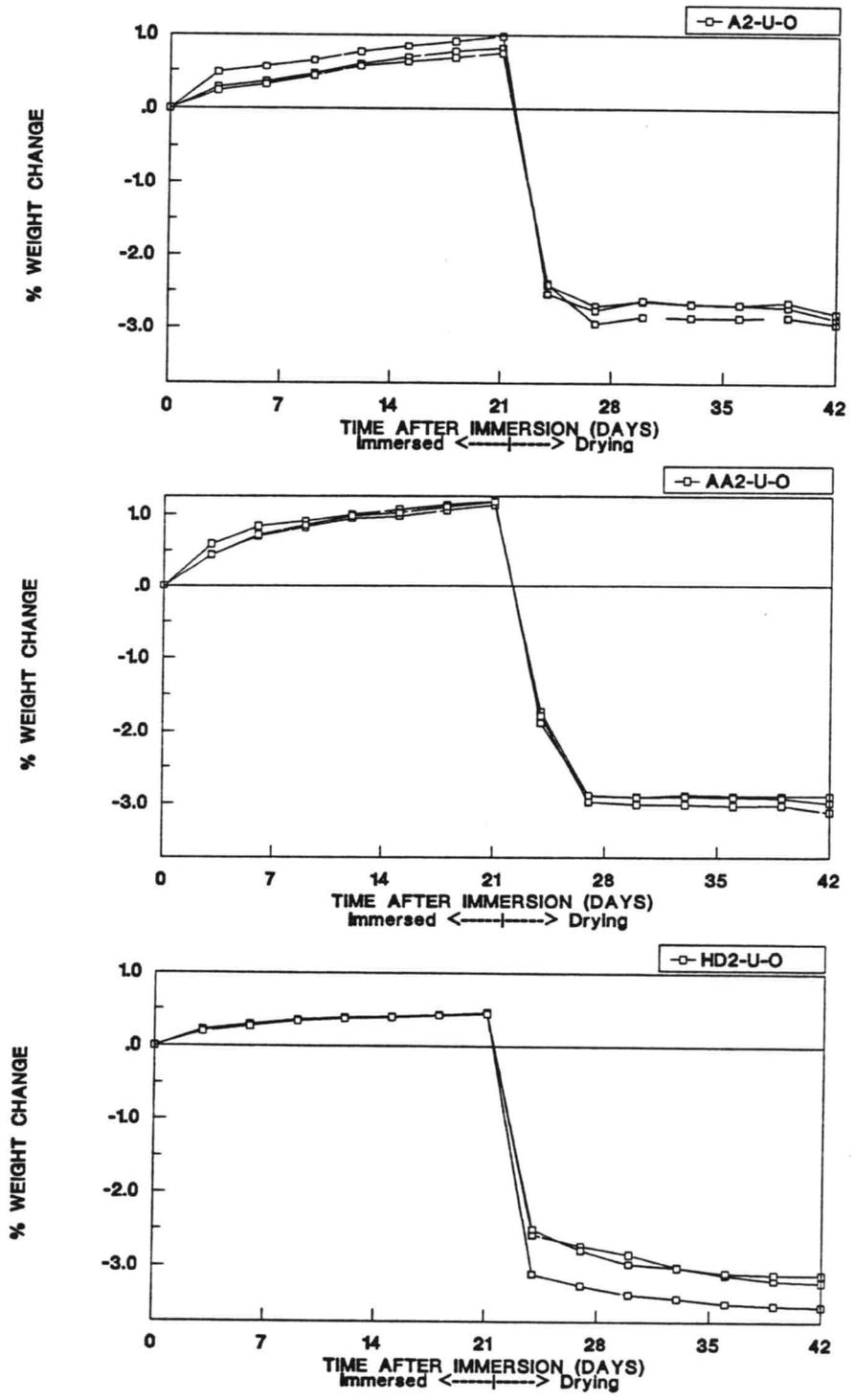


Fig. A.9 Batch #2, Untreated, Oven Dried, Phase I

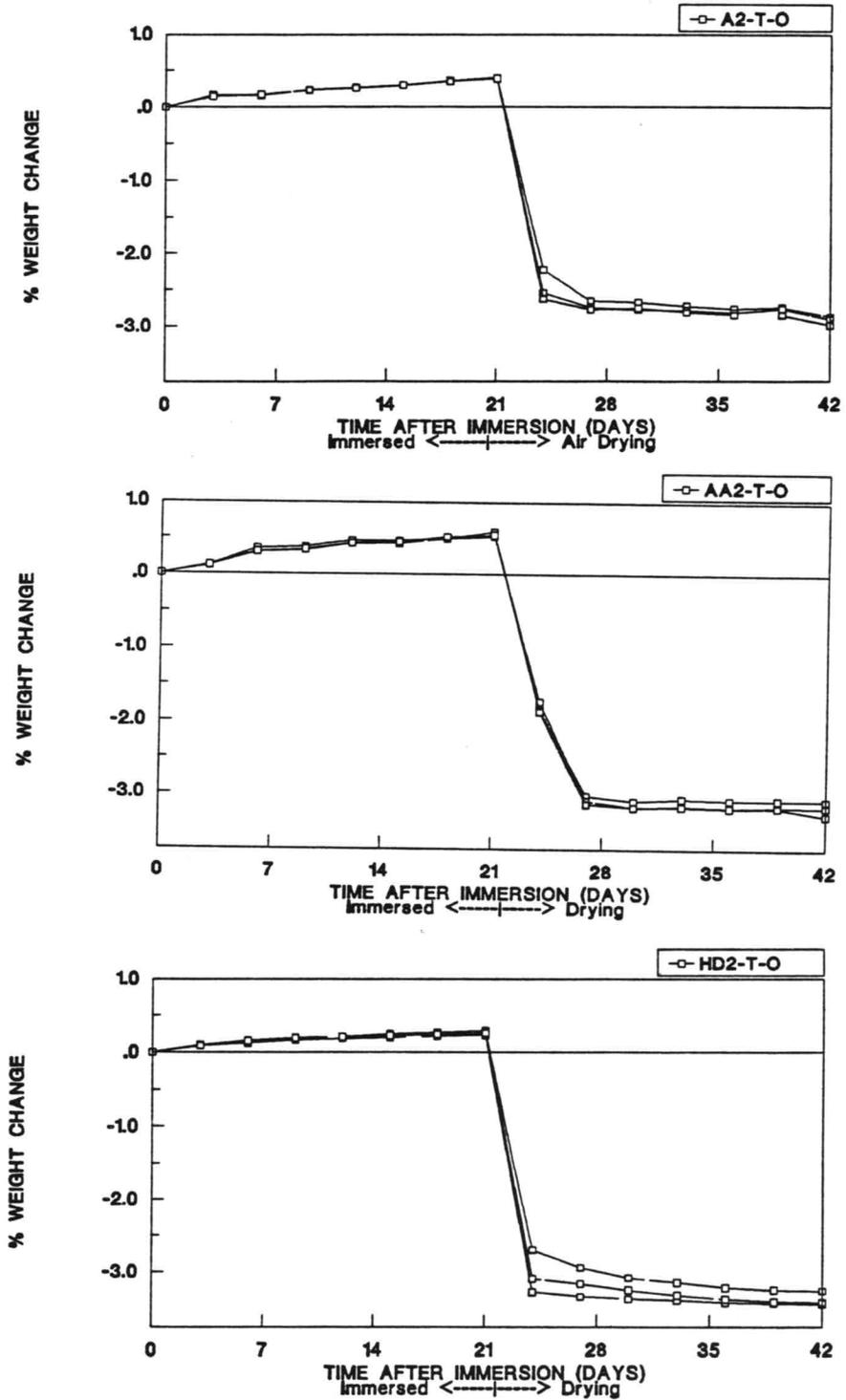


Fig. A.10 Batch #2, Treated, Oven Dried, Phase I

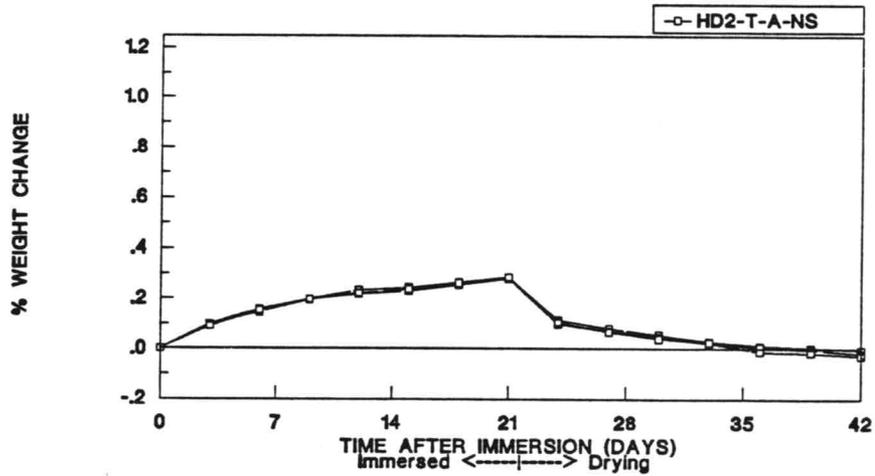
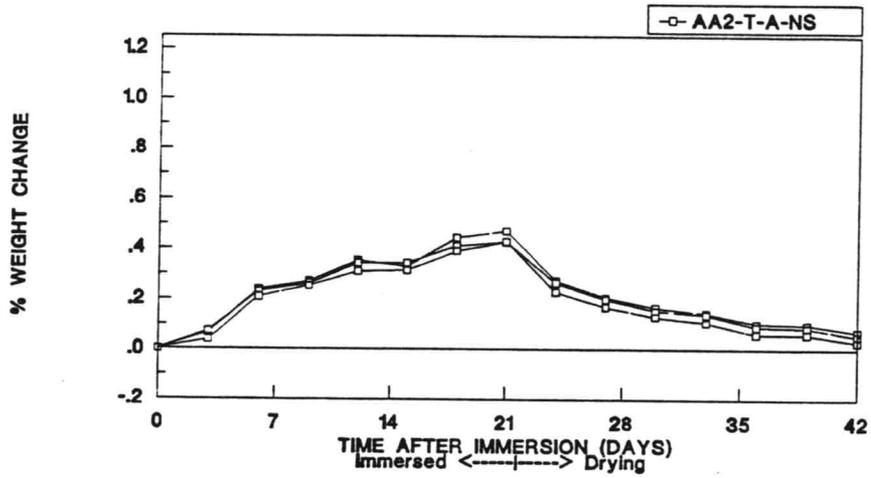
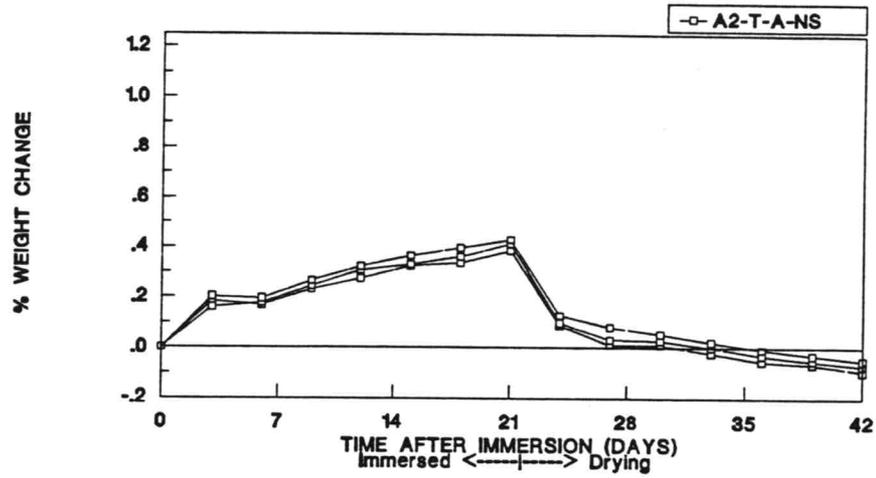


Fig. A.11 Batch #2, Treated, Air Dried, Not Sandblasted, Phase I

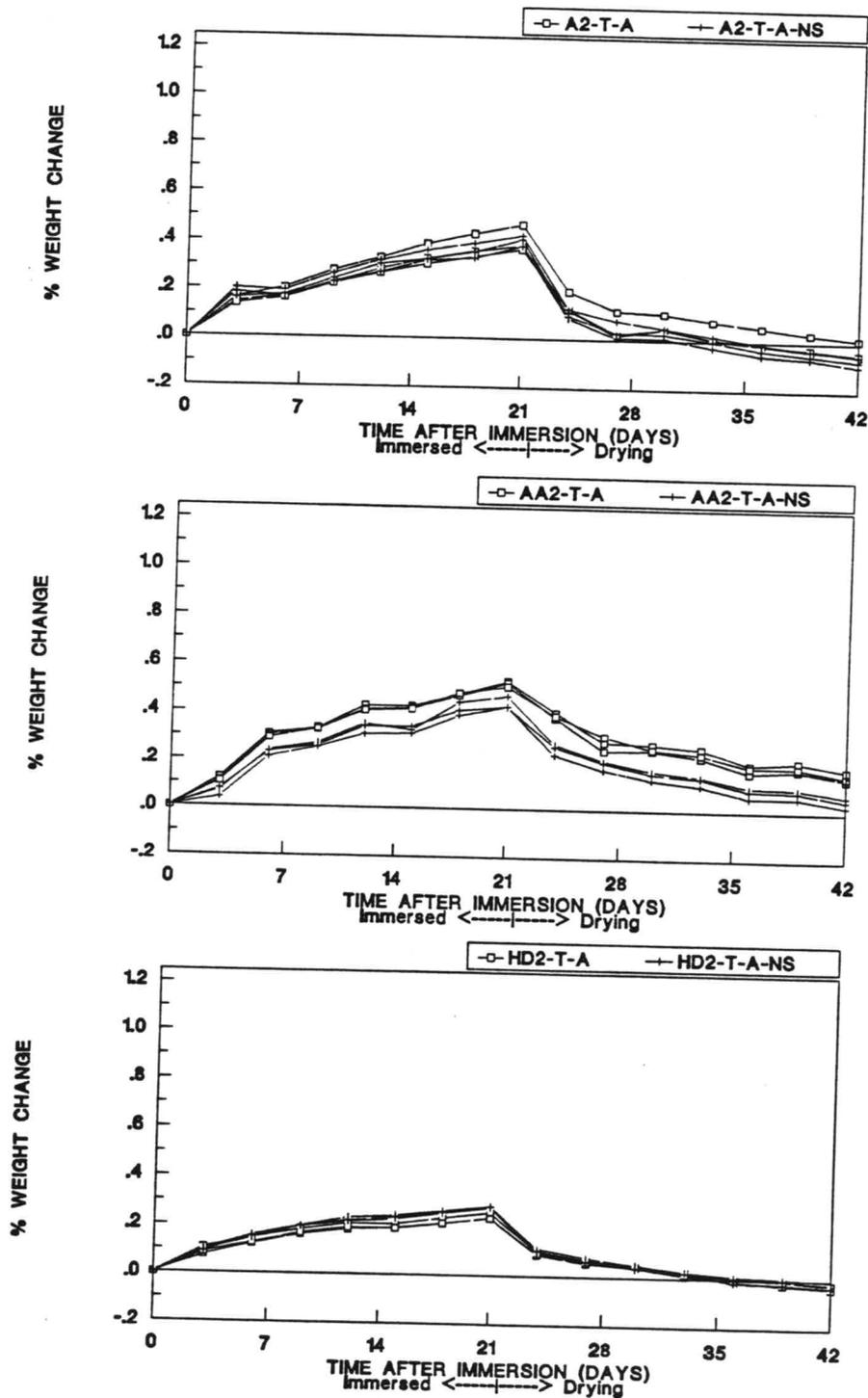


Fig. A.12 Batch #2, Treated, Air Dried, Sandblasted + Not Sandblasted, Phase I

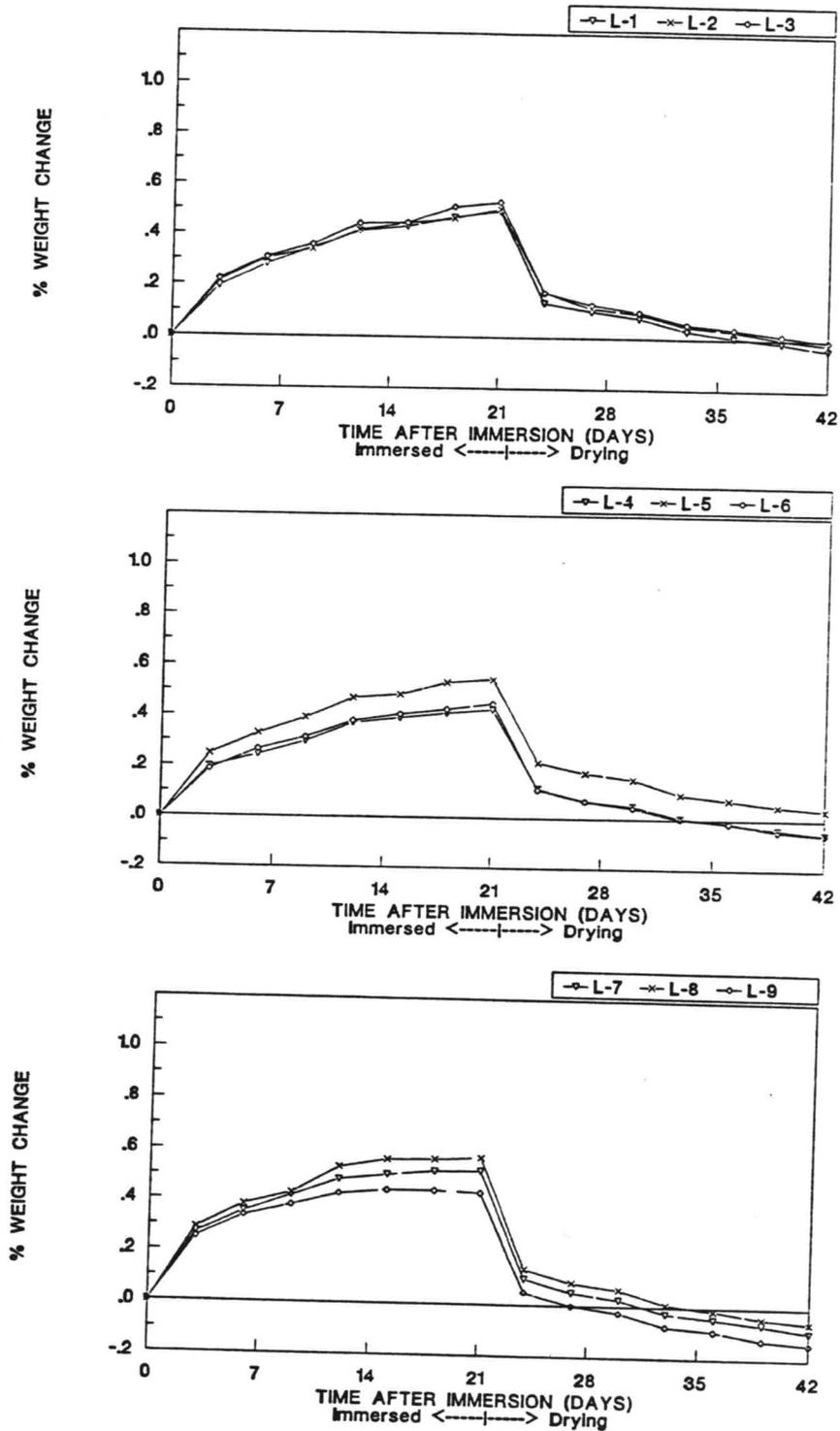


Fig. A.13 Linseed Cured, Phase III (T:shot-blast, M:water wash, B:no clean)

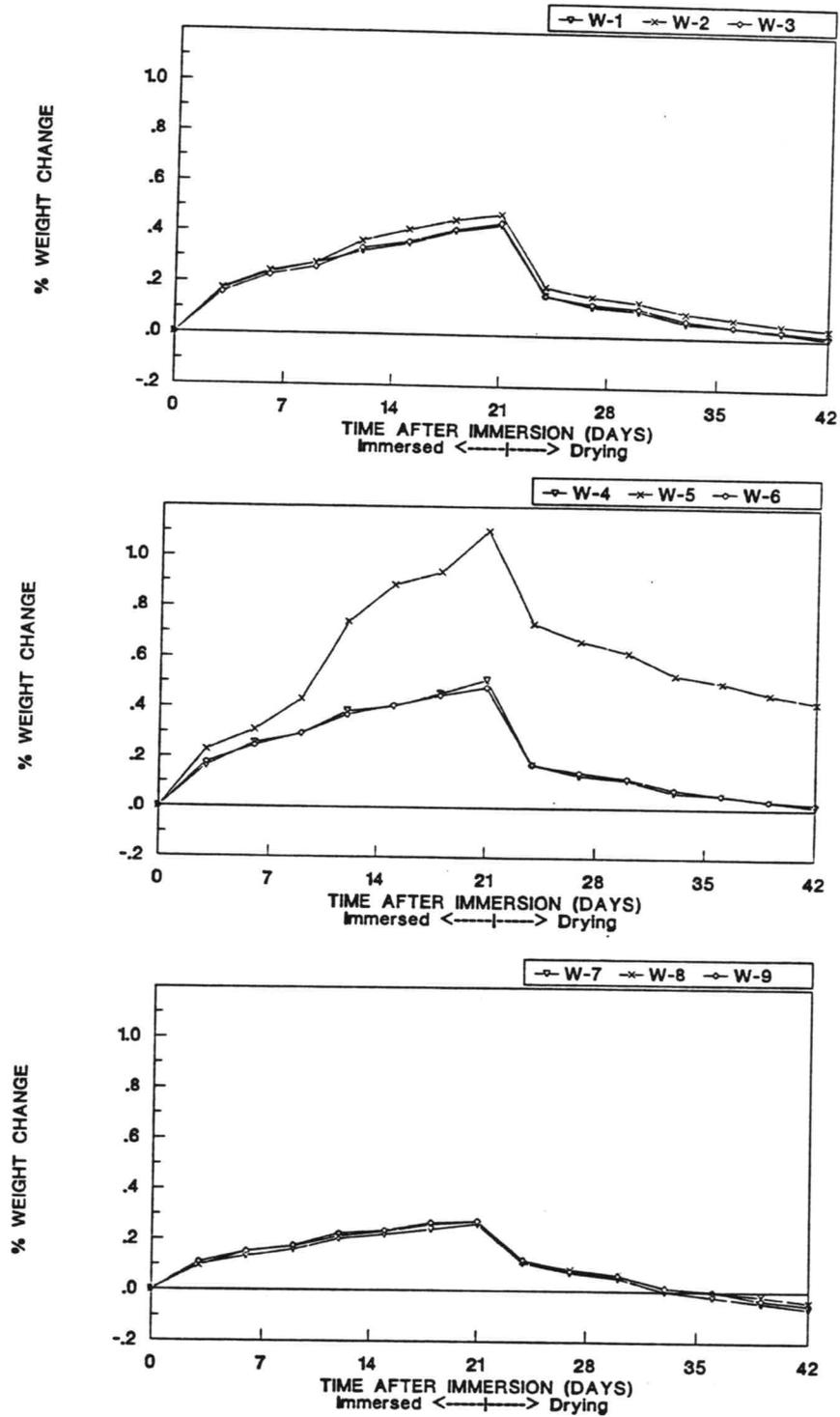


Fig. A.14 Membrane Cured, Phase III (T:shot-blast, M:water wash, B:no clean)

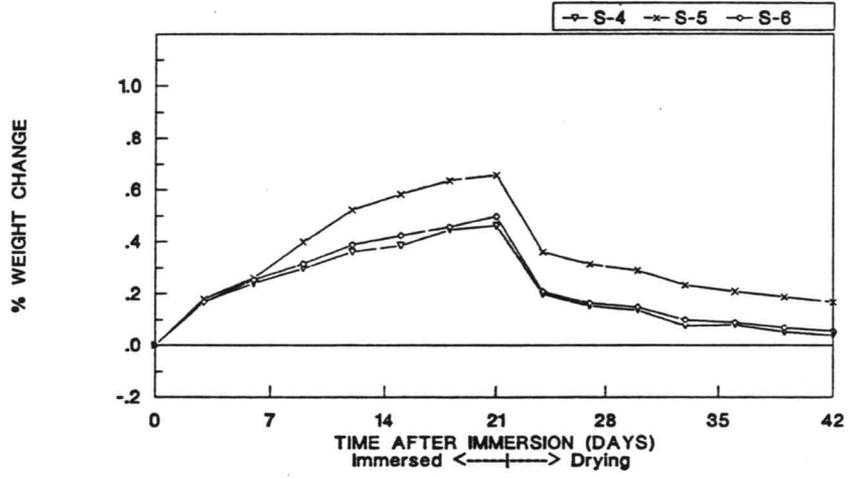
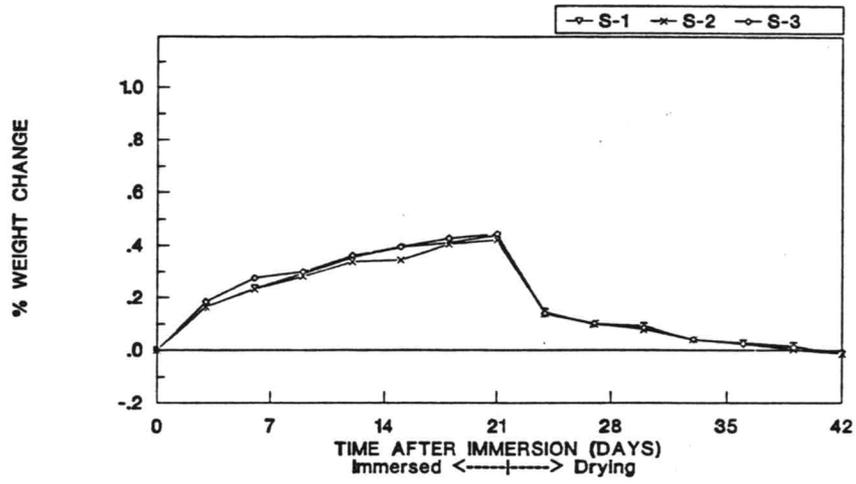


Fig. A.15 Self-Cured, Phase III (T:shot-blast, B:water wash)

A.2 CHLORIDE ANALYSIS DATA

Chloride analysis data is presented for AASHTO 259-80 and NCHRP 244 tests. Determination of the acid-soluble chloride ion concentration was performed in accordance with AASHTO T 260-80 for both tests. Presented are **total chlorides** in ppm, and in lb/cyd. Total chlorides at the corresponding depth of the control specimen must be subtracted to obtain absorbed chlorides. Specimen designations are included to indicate the concrete class, batch number, specimen number, hole number, and the depth level at which the sample was collected. Depth level 1 is 1.6 to 13 mm (1/16 to 1/2 in.), and depth level 2 is 13 to 25 mm (1/2 to 1 in.). All measurements refer to the top surface of the specimen.

For the Phase I and II AASHTO tests, specimen numbers 1-3 are treated, and specimen numbers 4-6 are untreated. The control specimen used for both Phase I NCHRP and AASHTO tests is specimen number 7 for each concrete class. Two control specimens were used for Phase II class HD concrete (specimens 7 and 8). For example: sample AA1-2-3-2 is a sample from class AA concrete first batch, specimen number 2 (treated slab), hole number 3, second depth.

Phase I NCHRP test specimens 28-30 are treated cube specimens, and specimens 34-36 are untreated. All specimens sampled in this test are air dried specimens. For example: sample HD1-29-2-1 is a sample from high density concrete first batch, specimen number 29 (treated cube), hole number 2, depth level 1.

A.2.1 Chloride Analysis Data, Phase I, AASHTO T259 Test

| Sample | | | Sample | | |
|-------------|----------|-------------|-------------|----------|-------------|
| Designation | Cl (ppm) | Cl (lb/cyd) | Designation | Cl (ppm) | Cl (lb/cyd) |
| A1-1-1-1 | 474 | 1.85 | AA1-1-2-2 | 360 | 1.41 |
| A1-1-1-2 | 401 | 1.57 | AA1-1-3-1 | 804 | 3.14 |
| A1-1-2-1 | 622 | 2.43 | AA1-1-3-2 | 333 | 1.30 |
| A1-1-2-2 | 346 | 1.35 | AA1-2-1-1 | 1100 | 4.30 |
| A1-1-3-1 | 546 | 2.13 | AA1-2-1-2 | 436 | 1.71 |
| A1-1-3-2 | 452 | 1.77 | AA1-2-2-1 | 853 | 3.34 |
| A1-2-1-1 | 426 | 1.66 | AA1-2-2-2 | 314 | 1.23 |
| A1-2-1-2 | 458 | 1.79 | AA1-2-3-1 | 1170 | 4.58 |
| A1-2-2-1 | 881 | 3.45 | AA1-2-3-2 | 374 | 1.46 |
| A1-2-2-2 | 495 | 1.94 | AA1-3-1-1 | 676 | 2.64 |
| A1-2-3-1 | 420 | 1.64 | AA1-3-1-2 | 280 | 1.09 |
| A1-2-3-2 | 270 | 1.06 | AA1-3-2-1 | 709 | 2.77 |
| A1-3-1-1 | 1280 | 5.01 | AA1-3-2-2 | 410 | 1.60 |
| A1-3-1-2 | 276 | 1.08 | AA1-3-3-1 | 572 | 2.24 |
| A1-3-2-1 | 874 | 3.42 | AA1-3-3-2 | 306 | 1.19 |
| A1-3-2-2 | 372 | 1.45 | AA1-3-3-2 | 295 | 1.15 |
| A1-3-3-1 | 982 | 3.84 | AA1-4-1-1 | 1540 | 6.03 |
| A1-3-3-2 | 427 | 1.67 | AA1-4-1-2 | 326 | 1.27 |
| A1-4-1-1 | 2350 | 9.20 | AA1-4-1-2 | 370 | 1.45 |
| A1-4-1-2 | 743 | 2.91 | AA1-4-2-1 | 921 | 3.60 |
| A1-4-2-1 | 2400 | 9.39 | AA1-4-2-2 | 415 | 1.62 |
| A1-4-2-2 | 407 | 1.59 | AA1-4-3-1 | 945 | 3.70 |
| A1-4-3-1 | 2510 | 9.83 | AA1-4-3-2 | 313 | 1.22 |
| A1-4-3-2 | 586 | 2.29 | AA1-5-1-1 | 478 | 1.87 |
| A1-4-4-1 | 3160 | 12.37 | AA1-5-1-2 | 341 | 1.33 |
| A1-4-4-2 | 678 | 2.65 | AA1-5-2-1 | 814 | 3.18 |
| A1-5-1-1 | 986 | 3.86 | AA1-5-2-2 | 485 | 1.90 |
| A1-5-1-2 | 309 | 1.21 | AA1-5-3-1 | 1710 | 6.69 |
| A1-5-2-1 | 504 | 1.97 | AA1-5-3-2 | 402 | 1.57 |
| A1-5-2-2 | 384 | 1.50 | AA1-6-1-1 | 1160 | 4.54 |
| A1-5-3-1 | 471 | 1.84 | AA1-6-1-2 | 352 | 1.38 |
| A1-5-3-2 | 298 | 1.16 | AA1-6-2-1 | 1900 | 7.44 |
| A1-6-1-1 | 592 | 2.31 | AA1-6-2-2 | 416 | 1.63 |
| A1-6-1-2 | 352 | 1.37 | AA1-6-3-1 | 1730 | 6.77 |
| A1-6-2-1 | 445 | 1.74 | AA1-6-3-2 | 334.5 | 1.31 |
| A1-6-2-2 | 467 | 1.83 | AA1-7-1-1 | 360 | 1.41 |
| A1-6-3-1 | 513 | 2.01 | AA1-7-1-2 | 320 | 1.25 |
| A1-6-3-2 | 327 | 1.28 | AA1-7-2-1 | 361.5 | 1.41 |
| A1-7-1-1 | 360 | 1.41 | AA1-7-2-2 | 377.5 | 1.47 |
| A1-7-1-2 | 368 | 1.44 | AA1-7-3-1 | 271.75 | 1.06 |
| A1-7-2-1 | 388 | 1.52 | AA1-7-3-2 | 356 | 1.39 |
| A1-7-2-2 | 421 | 1.65 | HD1-1-1-1 | 213 | 0.83 |
| A1-7-3-1 | 353 | 1.38 | HD1-1-1-2 | 89.1 | 0.35 |
| A1-7-3-2 | 426 | 1.66 | HD1-1-2-1 | 279 | 1.09 |
| AA1-1-1-1 | 464 | 1.82 | HD1-1-2-2 | 52.9 | 0.21 |
| AA1-1-1-2 | 302 | 1.18 | HD1-1-3-2 | 114 | 0.44 |
| AA1-1-2-1 | 392 | 1.53 | HD1-2-1-1 | 292 | 1.14 |

| Sample | | |
|--------------------|-----------------|--------------------|
| <u>Designation</u> | <u>Cl (ppm)</u> | <u>Cl (lb/cyd)</u> |
| HD1-2-1-2 | 75.6 | 0.29 |
| HD1-2-2-1 | 311 | 1.22 |
| HD1-1-3-1 | 260 | 1.02 |
| HD1-2-2-2 | 95 | 0.37 |
| HD1-2-3-1 | 292 | 1.14 |
| HD1-2-3-2 | 80.9 | 0.32 |
| HD1-3-1-1 | 477 | 1.86 |
| HD1-3-1-2 | 80.2 | 0.31 |
| HD1-3-2-1 | 280 | 1.09 |
| HD1-3-2-2 | 104 | 0.41 |
| HD1-3-3-1 | 548 | 2.14 |
| HD1-3-3-2 | 60.8 | 0.24 |
| HD1-4-1-1 | 668 | 2.61 |
| HD1-4-1-2 | 144 | 0.56 |
| HD1-4-2-1 | 937 | 3.67 |
| HD1-4-2-2 | 91.5 | 0.36 |
| HD1-4-3-1 | 965 | 3.78 |
| HD1-4-3-2 | 90.2 | 0.35 |
| HD1-5-1-1 | 369 | 1.44 |
| HD1-5-1-2 | 97.7 | 0.38 |
| HD1-5-2-1 | 440 | 1.72 |
| HD1-5-2-2 | 127 | 0.49 |
| HD1-5-3-1 | 538 | 2.11 |
| HD1-5-3-2 | 57.3 | 0.22 |
| HD1-6-1-1 | 421 | 1.65 |
| HD1-6-1-2 | 102 | 0.40 |
| HD1-6-2-1 | 319 | 1.25 |
| HD1-6-2-2 | 86.5 | 0.34 |
| HD1-6-3-1 | 422 | 1.65 |
| HD1-6-3-2 | 141 | 0.55 |
| HD1-7-1-1 | 85 | 0.33 |
| HD1-7-1-2 | 101 | 0.39 |
| HD1-7-2-1 | 112 | 0.44 |
| HD1-7-2-2 | 104 | 0.41 |
| HD1-7-3-1 | 101 | 0.39 |
| HD1-7-3-2 | 108 | 0.42 |

A.2.2 Chloride Analysis Data, Phase I, NCHRP 244 Test

| Sample | | | Sample | | |
|-------------|----------|-------------|-------------|----------|-------------|
| Designation | Cl (ppm) | Cl (lb/cyd) | Designation | Cl (ppm) | Cl (lb/cyd) |
| A1-28-1-1 | 571 | 2.23 | AA1-28-3-2 | 347 | 1.36 |
| A1-28-1-2 | 355 | 1.39 | AA1-28-4-1 | 901 | 3.53 |
| A1-28-2-2 | 435 | 1.70 | AA1-28-4-2 | 566 | 2.21 |
| A1-28-2-1 | 545 | 2.13 | AA1-29-1-1 | 427 | 1.67 |
| A1-28-3-1 | 655 | 2.56 | AA1-29-1-2 | 366 | 1.43 |
| A1-28-3-2 | 331 | 1.29 | AA1-29-2-1 | 395 | 1.54 |
| A1-28-4-1 | 462 | 1.81 | AA1-29-2-2 | 412 | 1.61 |
| A1-29-1-1 | 456 | 1.78 | AA1-29-1-3 | 336 | 1.31 |
| A1-29-1-2 | 416 | 1.63 | AA1-29-2-3 | 300 | 1.17 |
| A1-29-2-1 | 558 | 2.18 | AA1-29-3-1 | 702 | 2.75 |
| A1-29-2-2 | 379 | 1.48 | AA1-29-3-2 | 334 | 1.31 |
| A1-29-3-1 | 470 | 1.84 | AA1-29-4-1 | 609 | 2.38 |
| A1-29-3-2 | 407 | 1.59 | AA1-29-4-2 | 286 | 1.12 |
| A1-29-4-1 | 1120 | 4.38 | AA1-29-5-1 | 810 | 3.17 |
| A1-29-4-2 | 478 | 1.87 | AA1-29-5-2 | 426 | 1.66 |
| A1-29-5-1 | 378 | 1.48 | AA1-29-6-1 | 636 | 2.49 |
| A1-29-5-2 | 318 | 1.24 | AA1-29-6-2 | 387 | 1.51 |
| A1-29-6-1 | 315 | 1.23 | AA1-30-1-1 | 523 | 2.05 |
| A1-29-6-2 | 472 | 1.85 | AA1-30-1-2 | 306 | 1.19 |
| A1-30-1-1 | 403 | 1.57 | AA1-30-2-1 | 614 | 2.40 |
| A1-30-1-2 | 320 | 1.25 | AA1-30-2-2 | 347 | 1.36 |
| A1-30-2-1 | 535 | 2.09 | AA1-30-3-1 | 339 | 1.33 |
| A1-30-2-2 | 309 | 1.21 | AA1-30-3-2 | 337 | 1.32 |
| A1-30-3-1 | 526 | 2.06 | AA1-34-1-1 | 3070 | 12.02 |
| A1-30-3-2 | 445 | 1.74 | AA1-34-1-2 | 745 | 2.92 |
| A1-34-1-1 | 415 | 1.62 | AA1-34-2-1 | 1780 | 6.97 |
| A1-34-2-1 | 688 | 2.69 | AA1-34-2-2 | 265 | 1.04 |
| A1-34-2-1 | 596 | 2.33 | AA1-34-3-1 | 1900 | 7.44 |
| A1-34-2-2 | 387 | 1.51 | AA1-34-3-2 | 503 | 1.97 |
| A1-34-3-1 | 2270 | 8.88 | AA1-34-4-1 | 1760 | 6.89 |
| A1-34-3-2 | 832 | 3.25 | AA1-34-4-2 | 537 | 2.10 |
| A1-35-1-1 | 784 | 3.07 | AA1-34-5-1 | 1510 | 5.91 |
| A1-35-1-2 | 431 | 1.68 | AA1-34-5-2 | 333 | 1.30 |
| A1-35-2-1 | 860 | 3.36 | AA1-34-6-1 | 1120 | 4.38 |
| A1-35-3-1 | 484 | 1.89 | AA1-34-6-2 | 468 | 1.83 |
| A1-35-3-2 | 350 | 1.37 | AA1-35-1-1 | 1320 | 5.16 |
| A1-35-4-1 | 753 | 2.95 | AA1-35-1-2 | 299 | 1.17 |
| A1-35-4-2 | 348 | 1.36 | AA1-35-2-1 | 2310 | 9.04 |
| A1-36-1-1 | 758 | 2.96 | AA1-35-2-2 | 366 | 1.43 |
| A1-36-1-2 | 352 | 1.37 | AA1-35-3-1 | 1640 | 6.42 |
| A1-36-2-1 | 984 | 3.85 | AA1-35-3-2 | 436 | 1.71 |
| A1-36-2-2 | 329 | 1.28 | AA1-35-4-1 | 1410 | 5.52 |
| A1-36-3-1 | 655 | 2.56 | AA1-35-4-2 | 363 | 1.42 |
| A1-36-3-2 | 377 | 1.47 | AA1-35-5-1 | 851 | 3.33 |
| AA1-28-2-1 | 621 | 2.43 | AA1-35-5-2 | 333 | 1.30 |
| AA1-28-2-2 | 255 | 0.99 | AA1-35-6-1 | 1110 | 4.34 |
| AA1-28-3-1 | 914 | 3.58 | AA1-35-6-2 | 316 | 1.24 |

| Sample | | |
|--------------------|-----------------|--------------------|
| <u>Designation</u> | <u>Cf (ppm)</u> | <u>Cf (lb/cyd)</u> |
| AA1-36-1-1 | 1420 | 5.56 |
| AA1-36-1-2 | 274 | 1.07 |
| AA1-36-2-1 | 1660 | 6.50 |
| AA1-36-2-2 | 409 | 1.60 |
| AA1-36-3-1 | 1940 | 7.59 |
| AA1-36-3-2 | 714 | 2.79 |
| AA1-36-4-1 | 2740 | 10.73 |
| AA1-36-4-2 | 362 | 1.42 |
| AA1-36-5-1 | 811 | 3.17 |
| AA1-36-5-2 | 294 | 1.15 |
| AA1-36-6-1 | 1510 | 5.91 |
| AA1-36-6-2 | 424 | 1.66 |
| HD1-28-1-1 | 185 | 0.74 |
| HD1-28-1-2 | 95.8 | 0.37 |
| HD1-28-2-1 | 286 | 1.12 |
| HD1-28-2-2 | 151.5 | 0.59 |
| HD1-28-3-1 | 467 | 1.83 |
| HD1-28-3-2 | 318 | 1.24 |
| HD1-28-4-1 | 377 | 1.47 |
| HD1-28-4-2 | 126 | 0.49 |
| HD1-28-5-1 | 89.9 | 0.35 |
| HD1-28-5-2 | 124 | 0.48 |
| HD1-28-6-1 | 468 | 1.83 |
| HD1-28-6-2 | 181 | 0.71 |
| HD1-29-1-1 | 157 | 0.61 |
| HD1-29-1-2 | 124 | 0.48 |
| HD1-29-2-1 | 199 | 0.78 |
| HD1-29-2-2 | 140 | 0.55 |
| HD1-29-3-1 | 606 | 2.37 |
| HD1-29-3-2 | 108 | 0.42 |
| HD1-29-4-1 | 288 | 1.13 |
| HD1-29-4-2 | 119 | 0.46 |
| HD1-29-5-1 | 176.5 | 0.69 |
| HD1-29-5-2 | 109 | 0.42 |
| HD1-29-6-1 | 122 | 0.47 |
| HD1-29-6-2 | 147 | 0.57 |
| HD1-30-1-1 | 260 | 1.02 |
| HD1-30-1-2 | 239.8 | 0.94 |
| HD1-30-3-1 | 814 | 3.18 |
| HD1-30-3-2 | 549 | 2.15 |
| HD1-30-4-1 | 264 | 1.03 |
| HD1-30-4-2 | 80.7 | 0.31 |
| HD1-30-5-1 | 139 | 0.54 |
| HD1-30-5-2 | 138 | 0.54 |
| HD1-30-6-1 | 93.3 | 0.36 |
| HD1-30-6-2 | 103 | 0.40 |
| HD1-34-1-1 | 350 | 1.37 |
| HD1-34-1-2 | 139 | 0.54 |
| HD1-34-2-1 | 186 | 0.73 |

| Sample | | |
|--------------------|-----------------|--------------------|
| <u>Designation</u> | <u>Cf (ppm)</u> | <u>Cf (lb/cyd)</u> |
| HD1-34-2-2 | 109 | 0.42 |
| HD1-34-3-1 | 186 | 0.73 |
| HD1-34-3-2 | 173 | 0.67 |
| HD1-34-4-1 | 498 | 1.95 |
| HD1-34-4-2 | 45.2 | 0.17 |
| HD1-34-6-1 | 381 | 1.49 |
| HD1-34-6-2 | 111 | 0.43 |
| HD1-35-1-1 | 253 | 0.99 |
| HD1-35-1-2 | 112 | 0.44 |
| HD1-35-2-1 | 373 | 1.46 |
| HD1-35-2-2 | 123 | 0.48 |
| HD1-35-3-1 | 677 | 2.65 |
| HD1-35-3-2 | 526 | 2.06 |
| HD1-35-4-1 | 426 | 1.66 |
| HD1-35-4-2 | 162 | 0.63 |
| HD1-35-5-1 | 791 | 3.09 |
| HD1-35-5-2 | 125 | 0.49 |
| HD1-35-6-1 | 257 | 1.01 |
| HD1-35-6-2 | 114 | 0.44 |
| HD1-36-1-1 | 263 | 1.03 |
| HD1-36-1-2 | 140 | 0.55 |
| HD1-36-2-1 | 394 | 1.54 |
| HD1-36-2-2 | 119 | 0.46 |
| HD1-36-3-1 | 472 | 1.85 |
| HD1-36-3-2 | 140 | 0.55 |
| HD1-36-4-1 | 171 | 0.67 |
| HD1-36-4-2 | 92.5 | 0.36 |
| HD1-36-5-1 | 1030 | 4.03 |
| HD1-36-5-2 | 235 | 0.92 |
| HD1-36-6-1 | 675 | 2.64 |
| HD1-36-6-2 | 155 | 0.60 |

A.2.3 Chloride Analysis Data, Phase II, AASHTO T259 Test

| Sample | | | Sample | | |
|--------------------|-----------------|--------------------|--------------------|-----------------|--------------------|
| <u>Designation</u> | <u>Cl (ppm)</u> | <u>Cl (lb/cyd)</u> | <u>Designation</u> | <u>Cl (ppm)</u> | <u>Cl (lb/cyd)</u> |
| AA3-1-1-1 | 543 | 2.12 | HD3-1-3-2 | 79.3 | 0.31 |
| AA3-1-1-2 | 362 | 1.42 | HD3-2-1-1 | 549 | 2.15 |
| AA3-1-2-1 | 442 | 1.73 | HD3-2-1-2 | 118 | 0.46 |
| AA3-1-2-2 | 293 | 1.15 | HD3-2-2-1 | 616 | 2.41 |
| AA3-1-3-1 | 984 | 3.85 | HD3-2-2-2 | 89.5 | 0.35 |
| AA3-1-3-2 | 367 | 1.44 | HD3-2-3-1 | 878 | 3.44 |
| AA3-2-1-1 | 463 | 1.81 | HD3-2-3-2 | 135 | 0.53 |
| AA3-2-1-2 | 365 | 1.43 | HD3-3-1-1 | 1320 | 5.16 |
| AA3-2-2-1 | 985 | 3.86 | HD3-3-1-2 | 133 | 0.52 |
| AA3-2-2-2 | 340 | 1.33 | HD3-3-2-1 | 277 | 1.08 |
| AA3-2-3-1 | 1480 | 5.79 | HD3-3-2-2 | 90 | 0.35 |
| AA3-2-3-2 | 312 | 1.22 | HD3-3-3-1 | 1130 | 4.42 |
| AA3-3-1-1 | 721 | 2.82 | HD3-3-3-2 | 81.6 | 0.32 |
| AA3-3-1-2 | 421 | 1.65 | HD3-4-1-1 | 1040 | 4.07 |
| AA3-3-2-1 | 1050 | 4.11 | HD3-4-1-2 | 98.2 | 0.38 |
| AA3-3-2-2 | 456 | 1.78 | HD3-4-2-1 | 1680 | 6.57 |
| AA3-3-3-1 | 954 | 3.73 | HD3-4-2-2 | 166 | 0.65 |
| AA3-3-3-2 | 387 | 1.51 | HD3-4-3-1 | 739 | 2.89 |
| AA3-4-1-1 | 2300 | 9.00 | HD3-4-3-2 | 105 | 0.41 |
| AA3-4-1-2 | 716 | 2.80 | HD3-5-1-1 | 1200 | 4.70 |
| AA3-4-2-1 | 1960 | 7.67 | HD3-5-1-2 | 247 | 0.96 |
| AA3-4-2-2 | 646 | 2.53 | HD3-5-2-1 | 833 | 3.26 |
| AA3-4-3-1 | 2590 | 10.14 | HD3-5-2-2 | 157 | 0.61 |
| AA3-4-3-2 | 642 | 2.51 | HD3-5-3-1 | 895 | 3.50 |
| AA3-5-1-1 | 3430 | 13.43 | HD3-5-3-2 | 167 | 0.65 |
| AA3-5-1-2 | 666 | 2.61 | HD3-6-1-1 | 1020 | 3.99 |
| AA3-5-2-1 | 4950 | 19.38 | HD3-6-1-2 | 101 | 0.39 |
| AA3-5-2-2 | 962 | 3.76 | HD3-6-2-1 | 1640 | 6.42 |
| AA3-5-3-1 | 3830 | 14.99 | HD3-6-2-2 | 153 | 0.59 |
| AA3-5-3-2 | 744 | 2.91 | HD3-6-3-1 | 1450 | 5.67 |
| AA3-6-1-1 | 1200 | 4.70 | HD3-6-3-2 | 113 | 0.44 |
| AA3-6-1-2 | 372 | 1.46 | HD3-7-1-1 | 97.8 | 0.38 |
| AA3-6-2-1 | 1620 | 6.34 | HD3-7-1-2 | 92.5 | 0.36 |
| AA3-6-2-2 | 303 | 1.18 | HD3-7-2-1 | 121 | 0.47 |
| AA3-6-3-1 | 2660 | 10.41 | HD3-7-2-2 | 161 | 0.63 |
| AA3-6-3-2 | 889 | 3.48 | HD3-7-3-1 | 105 | 0.41 |
| AA3-7-1-1 | 409 | 1.60 | HD3-7-3-2 | 162 | 0.63 |
| AA3-7-1-2 | 363 | 1.42 | HD3-8-1-1 | 88.4 | 0.35 |
| AA3-7-2-1 | 387 | 1.51 | HD3-8-1-2 | 133 | 0.52 |
| AA3-7-2-2 | 379 | 1.48 | HD3-8-2-1 | 135 | 0.53 |
| AA3-7-3-1 | 362 | 1.42 | HD3-8-2-2 | 85.4 | 0.33 |
| AA3-7-3-2 | 349 | 1.36 | HD3-8-3-1 | 321 | 1.25 |
| HD3-1-1-1 | 158 | 0.62 | HD3-8-3-2 | 196 | 0.76 |
| HD3-1-1-2 | 105 | 0.41 | | | |
| HD3-1-2-1 | 165 | 0.64 | | | |
| HD3-1-2-2 | 50.9 | 0.20 | | | |
| HD3-1-3-1 | 245 | 0.96 | | | |