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Effect of Canadian Linseed Oil Based Concrete Preservatives
On
the Durability of Concrete in Hong Kong

A Thesis
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for the Degree of

MASTER OF SCIENCE

by Yuping Xie

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- Health and Environmental Sciences
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- Health Sciences
  - General 0566
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  - Dentistry 0567
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Executive Summary

This research program was designed to examine the effectiveness of Canadian linseed oil-based concrete preservatives on the concrete roads and structures in Hong Kong. The experimental works were conducted at the University of Manitoba and the University of Hong Kong simultaneously following the same schedule and test manual.

This paper summarizes the works conducted at the two universities during the period from September 1993 to August 1995. The work includes the design of a test manual for the program, design and construction of the test equipment for both universities to examine the behaviors of concrete under the similar environmental conditions as Hong Kong. The University of Hong Kong provided all concrete specimens for both universities.

The experimental program investigated the effect of four Canadian linseed oil-based concrete preservatives P1, P2, P3 and P4 on concrete specimens using two types of mix designs, normal concrete G30/20 and high strength concrete G45/20 typically used in the structures and roads in Hong Kong. It was designed to include salt spray, carbonation, quick ultra-violet artificial weathering, bond strength and dripping tests. A total of more than 500 concrete blocks or cores of various sizes were fabricated by each university as test specimens.

The program shows that the equipment designed for this program could be adequately used to examine the effectiveness of concrete preservatives.

The results show that all four concrete preservatives considered in this program have significant effect on enhancing the resistance of G30/20 concrete against salt spray
attack and carbonation. Resistance of G45/20 concrete to salt spray is also enhanced by the concrete preservatives. The results are used to rank the products. No conclusion can be made regarding the effect of the four preservatives on improving the carbonation resistance of G45/20 concrete since all the preserved and non-preserved concrete specimens show no evidence of carbonation reaction after the test.

All the preserved concrete surfaces change colors after exposing to intense ultraviolet ray. Carbonation tests were performed after 100, 200, 500, 1000, 2000 and 4000 hours of QUV tests. The results indicate that the protection effect of all preservatives to carbonation disappears after 500 hours of QUV test. The QUV weathering reduces the carbonation resistance of all preserved concrete specimens. The duration limit of QUV weathering for the preserved concrete to remain superior to the non-preserved concrete is less than 500 hours. The QUV weathering improves the salt spray resistance of non-preserved concrete. After up to 500 hours of QUV weathering, all preserved concrete specimens still demonstrate higher salt spray resistance than non-preserved ones.

Bond strength test results show that the preservatives reduces the bond strength between concrete and mortar.

Based the dripping test results, the four concrete preservatives are capable of penetrating through vertical, horizontally upward and horizontally downward concrete surfaces with smooth or rough finish.

The results obtained by the two universities are consistent. As a conclusion, Canadian concrete preservative P1 is recommended to be used in the concrete structures and roads in Hong Kong.
# Table of Contents

## CHAPTER 1. INTRODUCTION

1.1 General ................................................................. 1
1.2 Objective ............................................................... 2
1.3 Scope ................................................................. 2

## CHAPTER 2. LITERATURE REVIEW

2.1 Use of Linseed Oil as Concrete Sealer .................................................. 4
  2.1.1 Linseed oil formulation ....................................................... 5
  2.1.2 Performance of linseed oil-based products ........................................ 6
  2.1.3 The economical benefit ......................................................... 9
  2.1.4 Measurement of penetration depth .............................................. 10
2.2 Effect of Chloride Intrusion on Concrete Structures .................................. 11
  2.2.1 Deterioration of concrete due to chloride penetration .......................... 11
  2.2.2 Methods for Evaluating Chloride Penetration Effect ............................ 13
2.3 Carbonation of Concrete .......................................................... 15
  2.3.1 Carbonation reaction in concrete ............................................... 16
  2.3.2 Accelerated carbonation test .................................................... 18
  2.3.3 Determination of carbonation depth ........................................... 19
2.4 Accelerated Artificial Weathering Test ............................................... 21
2.5 Bond Strength between Concrete and Mortar .......................................... 22

## CHAPTER 3. MATERIALS

3.1 Concrete Preservatives .......................................................... 24
3.2 Cement, Aggregates and Admixture .................................................... 24
3.3 Concrete Specimens ............................................................... 26
3.4 Mortar ................................................................. 27

## CHAPTER 4. TEST PROCEDURES

4.1 Application of Preservatives .......................................................... 28
4.2 Curing and Cutting of the Test Specimens ............................................ 31
4.3 Salt Spray Test ............................................................. 33
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.4 Carbonation Test</td>
<td>38</td>
</tr>
<tr>
<td>4.5 Bond Strength Test</td>
<td>40</td>
</tr>
<tr>
<td>4.6 Quick Ultraviolet Artificial Weathering Test</td>
<td>42</td>
</tr>
<tr>
<td>4.7 Dripping Test</td>
<td>46</td>
</tr>
<tr>
<td><strong>CHAPTER 5. RESULTS AND DISCUSSION</strong></td>
<td>48</td>
</tr>
<tr>
<td>5.1 Application of Preservatives</td>
<td>48</td>
</tr>
<tr>
<td>5.2 Salt Spray Test</td>
<td>52</td>
</tr>
<tr>
<td>5.3 Carbonation Test</td>
<td>59</td>
</tr>
<tr>
<td>5.4 Bond Strength Test</td>
<td>64</td>
</tr>
<tr>
<td>5.5 Quick Ultraviolet Artificial Weathering Test</td>
<td>65</td>
</tr>
<tr>
<td>5.6 Dripping Test</td>
<td>77</td>
</tr>
<tr>
<td><strong>CHAPTER 6. CONCLUSIONS</strong></td>
<td>83</td>
</tr>
<tr>
<td><strong>CHAPTER 7. RECOMMENDATIONS FOR FUTURE WORKS</strong></td>
<td>85</td>
</tr>
<tr>
<td><strong>CHAPTER 8. REFERENCE</strong></td>
<td>87</td>
</tr>
</tbody>
</table>
List of Tables

Table 3-1. The Tested Concrete Preservatives .................................................. 24
Table 3-2. Manufacturer’s Test Report for the Cement Used ............................... 25
Table 3-3. Typical Sieve Analysis of Aggregates (% Passing) ............................ 25
Table 3-4. Concrete Mix Designs ......................................................................... 26
Table 4-1. Test Surface Used by the Two Universities ........................................ 31
Table 5-1. The Actual Coverage Rate Measurement ............................................ 50
Table 5-2. Technical Data for the Spray Operation .............................................. 51
Table 5-3. Drying and Absorption of the Preservatives on Concrete Surface .......... 51
Table 5-4. Results of Chloride Content Analysis in Salt Spray Test .................... 53
Table 5-5. Results of Chloride Content Analysis in Salt Spray Test .................... 54
Table 5-6. Results of pH Value Analysis Test ...................................................... 61
Table 5-7. Results of Carbonation Depth Measurement ....................................... 61
Table 5-8. Results of Bond Strength Test ............................................................ 64
Table 5-9. Results of Visual Inspection for Colour Change .................................. 68
Table 5-10. Results of Visual Inspection for Chalking ........................................ 68
Table 5-11. Results of Visual Inspection for Cracking ......................................... 69
Table 5-12. Results of Visual Inspection for Blistering ...................................... 70
Table 5-13. Results of Carbonation Depth Measurement after QUV Test ............ 71
Table 5-14. Results of Chloride Content Analysis after QUV and Salt Spray Tests (%) 71
Table 5-15. Results of 3-day Dripping Test for Horizontal Specimens .................. 78
Table 5-16. Results of 3-day Dripping Test for Vertical Specimens ..................... 78
Table 5-17. Results of 28-day Dripping Test for Vertical Specimens .................... 79
List of Figures

Figure 4-1. Application Site for the Vertical Specimens ............................................... 30
Figure 4-2 Application Site for the Horizontal Specimens ............................................. 30
Figure 4-3. The Salt Spray Test Setup ................................................................. 34
Figure 4-4. Inside View of the Salt Spray Chamber ...................................................... 37
Figure 4-5. The Carbonation Test Setup ................................................................. 39
Figure 4-6. Bond Strength Test Setup ........................................................................ 41
Figure 4-7. The QUV Artificial Weathering Tester with Specimen Holders ...................... 43
Figure 4-8 Locations of Cores for Dripping Test .......................................................... 47
Figure 5-1. Results of Chloride Content Analysis .......................................................... 55
Figure 5-2. Results of Chloride Content Analysis .......................................................... 55
Figure 5-3. Resistance to Chloride Penetration for ....................................................... 56
Figure 5-4. Resistance to Chloride Penetration for ....................................................... 56
Figure 5-5. Results of Chloride Content Analysis .......................................................... 57
Figure 5-6. Results of Chloride Content Analysis .......................................................... 57
Figure 5-7. Resistance to Chloride Penetration for ....................................................... 58
Figure 5-8. Resistance to Chloride Penetration for ....................................................... 58
Figure 5-9. Results of pH Value Analysis for .............................................................. 62
Figure 5-10. Results of pH Value Analysis for .............................................................. 62
Figure 5-11. Results of Carbonation Depth Measurement for Concrete G 30/20 .............. 63
Figure 5-12. Degree of Colour Change after QUV Test for Concrete G 30/20 ................. 72
Figure 5-13. Degree of Colour Change after QUV Test for Concrete G 45/20 ............... 72
Figure 5-14. Degree of Chalking after QUV Test for Concrete G 30/20

Figure 5-15. Degree of Chalking after QUV Test for Concrete G 45/20

Figure 5-16. 18-day Carbonation Depth after QUV Test

Figure 5-17. Chloride Content for G30/20 Concrete after QUV and Salt Spray Tests

Figure 5-18. Chloride Content for G45/20 Concrete after QUV and Salt Spray Tests

Figure 5-19. Chloride Content Ratio for G30/20 Concrete after QUV and Salt Spray Tests

Figure 5-20. Chloride Content Ratio for G45/20 Concrete after QUV and Salt Spray Tests

Figure 5-21. Dripping Test Results for Horizontal Downward Specimens

Figure 5-22. Dripping Test Results for Horizontal Downward Specimens

Figure 5-23. Dripping Test Results on Vertical Specimens

Figure 5-24. Dripping Test Results on Vertical Specimens

Figure 5-25. Dripping Test Results on Vertical Specimens
CHAPTER 1. INTRODUCTION

In a 3-year research program conducted at the University of Manitoba [1], the Canadian linseed oil was proved to be one of the most effective sealants in reducing chloride intrusion, salt water absorption and surface scaling of concrete.

Hong Kong, as reported by Brian Wilkins [2], has a tropical monsoon climate with a mean relative humidity of 78%, mean air temperature of 22.8°C and mean annual rainfall of 2224.7 mm. It is also a marine environment suffering high concentrations of industrial and automotive emissions into the atmosphere. In 1985, it was announced that 577 out of the 1000 Housing Authority’s blocks had concrete problem. The primary destructive agents were chlorides, sulphates and carbon dioxide in combination with water. In recent years, a cement render with some kind of applied finish such as glass mosaic tiles is used to provide additional weathering protection qualities of an external wall, which on the other hand, brings an important problem of bonding between concrete and mortar.

1.1 General

The present research program was designed to examine the effectiveness of several Canadian products as concrete preservatives for concrete roads and structures in Hong Kong. The products were based on Canadian linseed oil and admixture specially designed to provide the preservatives, which could be sprayed on the concrete surface, with the
capability to enhance the serviceability and durability of the concrete from severe environmental conditions such as the conditions in Hong Kong.

1.2 Objective

The primary objective of this study was to evaluate the effectiveness of several Canadian products as concrete preservative for concrete roads and structures in Hong Kong. The work included extensive discussions with, and approval of, the Hong Kong authorities, design of the testing manual, and design and construction of all test equipment needed to examine the potential effectiveness of the various Canadian concrete preservatives. Future work will include both field test and laboratory test using fewer number of products based on the test results of the first phase.

1.3 Scope

The laboratory investigation is conducted at the University of Manitoba and the University of Hong Kong simultaneously using different surface finishes of concrete specimens from the same concrete mix designs. The concrete mixes are typically used by the housing and highway authorities of Hong Kong. The following tests are performed using both preserved and non-preserved concrete specimens:

(i) Salt spray
(ii) Carbonation
(iii) Debonding effect
(iv) Ultra-violet artificial weathering effect
(v) Dripping effect during and after application of the preservatives
All the test procedures are specifically designed to simulate the environmental conditions in Hong Kong.
CHAPTER 2. LITERATURE REVIEW

Based on the objective and scope of this program, an extensive literature survey was conducted to achieve a better understanding of the behaviors of linseed oil based products as concrete preservatives and the deterioration of concrete structures due to chloride intrusion and carbonation. Effort was also made, through the survey, to search for proper test methods for evaluation of the effect of sealer penetration, chloride penetration, carbonation and weathering on concrete and concrete structures. The test methods for evaluation of bond strength between concrete and mortar were also searched. This section provides a review of the literature survey.

2.1 Use of Linseed Oil as Concrete Sealer

Review of the literature indicates that boiled linseed oil is one of the most widely used sealer materials for protection of concrete structures [3]. In an investigation involving 30 selected concrete protective sealants, linseed oil proved superior to all other products in protecting concrete against surface scaling [4]. Application of linseed oil/mineral spirit mixtures or linseed oil emulsions to exposed bridge surfaces, in an attempt to protect the concrete from scaling, spalling and cracking caused by hot wind, freeze-thaw conditions and deicing chemicals, has been specified as a standard practice by a number of highway and transportation authorities in North America, including Oklahoma Highway Department [5][6], Pennsylvania Department of Highway [4][7], Texas State Department of Highway and Public Transportation [8], Ontario Department of Highway [9] and Illinois Department of Transportation [10] etc..
2.1.1 Linseed oil formulation

Linseed oil is a yellowish drying oil extracted from flax. It contains a large proportion of glycerides [5]. It can be used in the form of either solution or emulsion. The solution consists of linseed oil and mineral spirit with the proportional ratio of linseed oil to mineral spirit ranging from 90:10 to 30:70 [11][12][13], among which the most commonly used one is the solution of 50% linseed oil and 50% mineral spirits [14]. To accelerate the drying of the oil, the solution often contains dryers such as cobalt, manganese, lead salts or napthenic acids [14]. The penetration depth of the mixtures increases as the percentage of mineral spirits increases [13]. Another form of linseed oil solution consists of 50% linseed oil and 50% kerosene [8][9]. Linseed oil emulsions is composed of equal volume proportion of oil phase and water phase. The most emulsifiable oil phase is made of 96.4% boiled linseed oil, 3% saturated tallow alcohol and 0.6% of 2-amino-2-methyl-1-propanol. The water phase consists of 99.6% water, 0.37% sodium hydroxide and 0.03% dipicolinic acid [15]. There is no significant difference between the performance of emulsion-type linseed oil and that of a boiled linseed oil/mineral spirit solutions [15] or linseed oil/kerosene solutions [9]. Emulsions create fewer fire hazards and permit easier cleaning of the equipment used in spraying when compared with solutions [15].
2.1.2 Performance of linseed oil-based products

The use of linseed oil as curing and antispalling agents was a big step toward a solution to the perplexing problem of severe cracking and the subsequent deterioration of bridge decking. The linseed oil curing compound used by the Oklahoma Highway Department in 1970s [5] [6] could be applied to moist concrete almost immediately after pouring. It eliminates much of the hairline cracking frequently associated with more conventional curing methods. It also reduces the spalling effect of winter de-icing salts. Application of linseed oil to bridge decks that have been originally cured by other process proves beneficial too. Although spalling and cracking may have began, the application of linseed oil slows down the deterioration. According to the results obtained by Cecil H. Best and James F. Crary [16], boiled linseed oil spirits is an effective antiscaling agent roughly in proportion to the concentration of linseed oil. The presence of a wax-based or resin-based curing compound does not impair the effectiveness of boiled linseed oil in mineral spirits as an antiscaling compound. Periodic treatment with boiled linseed oil in mineral spirits can resist salt-scaling indefinitely and increase abrasion resistance significantly. After studying 141 bridge decks covering the ages from new to 10 years old at initial treatment of linseed oil antiscaling compound, D. D. Fowler and R. H. Mitchell [10] concluded that the onset of scaling could be delayed by linseed oil treatment. The antiscaling compound is more effective in delaying the onset of scaling than in controlling scaling once started. An interval of 4 years between treatment was recommended.

Linseed oil based products are also reported to be effective in reducing chloride penetration [12], since the impregnation of linseed oil mixtures reduces the total porosity
and changes the pore size distribution of hardened concrete [11]. J. W. Ballew and K. L. Highlands [7] compared six different proprietary bridge deck sealants with a linseed oil product for their effectiveness to protect concrete from chloride intrusion. The six coatings and linseed oil were placed in individual sections of a four-span continuous steel I-beam bridge. Cores were taken from the travel lane in each section tested approximately four months, one year, two years and four years after application of the sealants. Both chloride content at a depth of two inches in the deck and the permeability of the concrete surface were measured. It was found that none of the six sealants consistently performed better than linseed oil through the course of the study although all the proprietary sealants were far more expensive than linseed oil. In a recent investigation to assess the ability of the sealers to protect the embedded reinforcing steel from corrosion [8], the following classes of sealers were evaluated: silane and siloxanes, water-based epoxy, polyester, aluminum stearate, silicate and linseed oil, using specimens from both standard bridge deck concrete and mortar from type I cement and sand. The silane and siloxanes group performed best in all testing and linseed oil performed nearly as well. A similar result was obtained by J. Wright et. al.[1], indicating that linseed oil and siloxane sealers were equally effective and were both more effective than silane. However, in an investigation over a 5 year old bridge deck subjected to yearly applications of a linseed oil and mineral spirits compound [17] and exposed to a chloride environment, it was noticed that the amount of chloride at the level of reinforcing steel and the electrical potential measurements of the deck still kept rising with time, indicating that the protection
provided by the linseed oil/mineral spirit compound was sufficient to prevent the deterioration of a concrete deck.

Loss of friction is another issue of concerns related to linseed oil-treated concrete pavements. S. N. Runkle [18] examined six selected test sites with 9 different road conditions, using a skid test car and a skid test trailer. On dry pavements, skid resistance recovers almost completely in 4 to 6 hours after the treatment of linseed oil antispalling compounds. In the case of two-shot treatments, significant decrease in skid resistance occurs after each treatment. Although recovery was not complete until 6 hours after the treatment, it is 92 percent complete after only 2 to 3 hours for both treatments, which is in consistence with Kubie, Gast and Cowan [19]. On wet pavements, the loss and recovery of skid resistance varies greatly depending principally on the surface texture and cleanliness, application amount and the surface temperature at the time of application. Under the worst condition, the skid resistance drops to a dangerous level and remained there for 2 days. Although the decrease and early recovery are different for different conditions, the time required for full recovery appeared all the same, 8 to 9 days. For up to 3 days, the delay time between first and second treatment has little effect on the skid resistance after the second treatment.

The impregnation of linseed oil may also have influence on the compressive strength of concrete. According to Blankenhorn, Barnes, Kline and Cady [20], the variations in compression strength as a function of time after impregnation represents somewhat anomalous wanderings of the test data. However, it appeared that linseed oil
impregnation could affect the compressive strength of concrete in a predictable manner over a time scale of up to 120 days.

2.1.3 The economical benefit

As one of the penetrating sealers, the most widely use of linseed oil is on concrete bridge decks [21]. Paul D. Carter [22][23] clarifies that bridge concrete generally deteriorates at a rate requiring some form of rehabilitation within 20 to 25 years. By slowing down the rate of corrosion, freeze-thaw microcracking, surface scaling, alkali-aggregate reaction and other problems, sealers can prolong the service life of bridge deck concrete and possibly eliminate rehabilitation within the functional life span. As more concrete is sealed, unit costs will go down, since the life cycle costs of sealing are much lower than those of rehabilitation. High quality concrete, when sealed at an early age, may last indefinitely. Poor quality concrete, even when experiencing advanced stages of deterioration, may be seal to temporally prospone the time of failure necessitating replacement or repair. However, the serviceability of the penetrating sealants is significantly reduced when they are applied to either normally air entrained concrete shortly after initial signs of salt scaling have commenced or to non-air entrained concrete at various degrees of prior scaling. Maximum serviceability is achieved when the sealants are applied to new concrete suffices that have not been exposed to deicing salts [24]. Sealants should not normally be applied to adequately cured and air entrained concrete that meets the strength requirements and that has sufficient cover over the steel [25].
2.1.4 Measurement of penetration depth

P. D. Stewart et. al. [4] evaluated successfully the penetration depths of 30 selected sealants including linseed oil, epoxy resins, coal tar oils etc. to concrete by using a product called “Blak-Rag” to detect the presence of the sealants in trace amount. The product is a very low viscosity liquid of organic origin with an odor resembling ether. It is fluorescent under ultraviolet light and is soluble in all products tested. During the test, eight drops of “Blak-Ray” are added to 25 ml of each penetrating sealant. After completely stirring, the sealants are applied to the finished surface of concrete specimens. Allowing three days of drying, the concrete specimens are broken into two. The sections are placed under a binocular microscope. An ultraviolet light was directed on the samples and as the microscope picks up a trace of fluorescence, the distance between the fluorescence and the treated surface of the concrete specimen can be measured as the penetration depth.

L. E. Gast [26] prepares concrete block in polyethylene dishes of 4” x 4” x 1/8”. A stainless steel wedge of 4” x 3/4” is placed in the center of each dish to produce a weak area along which the block could be latter easily broken. Four days after the treatment of linseed oil, the concrete blocks were broken and the penetration of oil into concrete could be seen as a faint gray-yellow area on the broken side of the blocks. However, a better method for determining penetration is to spray the broken side of the block with a 50% sulfuric acid solution and bake in an oven at 130°C for 30 minutes. The acid treatment chars the organic material and produced a black area where the oil has penetrated.
In the sealer penetration test conducted by William Cura [8], the sealed specimens were cured for 7 days in the lab and then broken with a hammer. The broken surface was briefly soaked in water to darken the portion beyond the reach of sealers due to wetting. The depth of unwetted layer was measured as penetration depth. A similar method was used by J. Wright et al. [1].

2.2 Effect of Chloride Intrusion on Concrete Structures

Penetration of chloride ion through concrete is an important process governing the durability of reinforced and prestressed concrete structures. This section reviews the deterioration of concrete structures caused by chloride attack and the test methods used for evaluating chloride penetration effect.

2.2.1 Deterioration of concrete due to chloride penetration

The major factor in causing the corrosion of steel in reinforced and prestressed member has been well identified as the chloride ion and its role in destroying the passivity of iron ion in a concrete environment [27][28][29][30]. J. A. Qazweeni and O. K. Daoud [31] investigated a 20-year-old building suffering from concrete deterioration and concluded that the direct cause of failure was an combined action between chloride attack and carbonation.

In northern area, the corrosion is due to the penetration of solutions containing deicing salts through concrete cover and anchorage zones. In southern area, the source of
corrosion is marine salt sprayed directly into concrete structural element by wave or rain action [32][33].

S. C. Patodiga [34] summarizes the major causes of corrosion as: (a) carbonation of concrete and (b) influence of aggressive substance. There are three phases in the corrosion process: (a) chloride penetration; (b) corrosion in the presence of concrete cover and (c) free corrosion after loss of cover. When the pH value goes below 9 due to carbonation, the passive protective layer around the reinforcing bar gets disintegrated and corrosion process sets-in. In concrete structures of average quality and pH level above 9, a limiting chloride ion content of 0.15% by weight can start the process of corrosion. The pressure due to corrosion of reinforcement would cause disbondment and cracking of cover concrete, depending on the bar/concrete bond, the surrounding concrete, sizes of flaws within these system components and geometric parameters [35]. Upon loss of concrete cover, the corrosion process would be significantly accelerated.

As observed by Y. Paul Virmani [32], corrosion-related distress of reinforced concrete occurs most often in bridge element adjacent to leaking joints and faulty drains. Distress is initiated due to the corrosion of prestressing stands, steel anchor plates and conventional reinforcement. Corrosion of tendons in pre- and post-tensioned bridge components can result in failure of the steel and consequently the structure of a bridge. In some instances, corrosion-related failure of a structure may occur suddenly without sufficient warning. For ordinary Portland cements it appears to be a question of time before detrimental amounts of chloride reach embedded steel even through a high quality of concrete. For instance, at a depth of 20 mm below the surface, there was hardly any
chloride at six months of exposure, while after 12 months and 24 months the chloride reached about 0.13% and 0.3% respectively [36]. O. Al-Amoudi and M. Maslehuddin [37] immersed specimens of 31 x 31 x 152 mm with 12.5 mm concrete cover over 6 mm steel bar to 15.7% chloride solution. It was found that the corrosion of steel was initiated in approximately 20 days.

Other than the corrosion of steel reinforcements, the presence of chloride ions seems to have little effect on the degradation of hardened cement paste [38]. A recent study [39] on cement pastes after up to 3 years of immersion in chloride solution reveals that chloroaluminate hydrates is formed as soon as chloride penetrates the matrix. However, the relative amount of chloroaluminate hydrates formed does not seem directly related to the total amount of chlorides bound to the solid. In addition to the formation of chloroaluminate crystals, chloride ions tend to bond to the calcium-silicate-hydrate phase. The influence of chloroaluminate hydrates on the performance of cement and concrete is hardly found in literature.

Corrosion is caused by simultaneous presence of the three factors: water, water soluble salt and oxygen. It can be prevented by effectively eliminate at least one of them [34].

2.2.2 Methods for Evaluating Chloride Penetration Effect

One of the most commonly used method for rapid evaluation of chloride penetration in concrete is ASSTO T277-83 “Rapid Determination of the Chloride Permeability” [8][25][40][41]. According to this method, a thick (usually 50 mm) concrete disc is placed between two electrodes (usually copper meshes) in an arrangement
similar to that of a diffusion cell. Sodium chloride solution of 3% by weight is added to the chamber housing one side of the specimen and 0.1 M sodium hydroxide is added to the chamber housing the other side of the specimen. An electrical field of 60 V as recommended is applied between the electrodes and the amount of coulombs along 6 hours of testing are recorded. The test defines that a higher amount of coulombs represents a higher permeability of the concrete to chloride. Requiring only 6 hours to obtain the results, this standard test method is fast and simple. However, a strong controversy has been promoted [41] since the total current passing across the cell is a function of the amount and type of ions, but not of the chloride flux or chloride mobility.

Another test method recommended by the American Association of State Highway Officials is AASHTO T259-80 “Standard Test for Chloride Ion Intrusion into Hardened Concrete” [1][42]. Using this method [1], a 4% sodium chloride solution is placed on the top surface of a 305 x 305 x 76 mm concrete specimens and maintained at a depth of approximately 13 mm. The specimens are stored in this manner for a total period of 90 days, after which the solution is removed, the specimens are dried for 7 days and the top 2 mm layer of concrete is removed with a precision grinder. Another 6 mm thick top layer of concrete is then cut from each specimen and used as sample for chloride content analysis. A low percentage of chloride indicates a high resistance to chloride penetration.

To assess concrete performance under arctic condition, M. Monkwa et. al. [43] proposed a freeze-thaw test for concrete in sea water. Concrete specimens of 75 x 75 x 350 mm are frozen in cold air at -25°C for 16 hours and thawed in sea water at -1°C for 8 hours to simulate the conditions of the tidal zone. The length change of the specimens
instrumented with a vibrating wire is measured at the end of each thawing and freezing period, whereas the length of the other specimens was measured monthly.

In subtropical area, the wet-dry cycling is one of the dominant climate conditions. Hong Kong Specification for Civil Engineering Works [44] specifies a test method based on 4 hours salt water spray at 40°C followed by 8 hours drying at 40°C and 30% R.H. for a total of 1000 hours. The salt water is made up with 30 ± 5 g/litre sodium chloride salt in distilled water, correspondent to 3% by weight sodium chloride solution. Upon completion of the wet-dry cycling, concrete samples are taken from the test surface at every 5 mm depth increment for chloride content analysis. A low percentage of chloride indicates a high resistance to chloride penetration.

D. E. Gjørv and Ø. Vennesland [36] also proposed a test method for determination of chloride penetration in cement mortar. In this method, mortar specimens of 25 mm in diameter and 100 mm in length are produced and embedded in a 5 mm thick layer of epoxy. One end of each specimen is cut leaving a bulk surface free for exposure. After two to three months of water curing, the specimens are exposed to fresh circulating sea water at 7± 2 ° C and 20 ± 1° C alternatively. Upon finish of the exposure, chloride penetration is determined on 3 - 5 mm thick slices successively cut off from the specimens.

### 2.3 Carbonation of Concrete

Carbonation of concrete is one of the most threatening phenomena related to corrosion of rebars. Due to the lowering of pH in the carbonated concrete, the passivation layer of the steel is destroyed quickly and the corrosion process can develop freely [45]. In
a recent survey to estimate the extent of carbonation in buildings in Toronto [46] over a total of 134 public and residential buildings, it is concluded that at least a proportion of the building stocks would experience carbonation corrosion damage within a desired service life.

2.3.1 *Carbonation reaction in concrete*

During carbonation process, CO₂ in the air react with Ca(OH)₂ in concrete pore solutions to form CaCO₃ plus water, reducing the pH value of concrete from above 12 to about 8.3 [47]. The pH level of the aggressive solution is the most important factor controlling the durability of the normal and high performance cement pastes subjected to chemical attack. The total porosity and the depth of calcium leaching is found to increase with the decrease of the pH level [38]. The calcium silicate hydrates (C-S-H) in hardened cement paste being decomposed through carbonation to be altered into CaCO₃, SiO₂ and H₂O have been occurring widely in various types of concrete structures [49]. By carbonation with excess water, ettringite is clearly decomposed to gypsum, calcium carbonate and aluminum gel [51]. E. Revertegat et al. [49] immerses specimens of neat cement paste in solution with different pH values for a total of 3 years. With pH value of 13, decalcification is barely seen. With pH value of 11.5, the total lime content drops by 35% for ordinary Portland cement. With pH value of 4.5, the decalcification ranged from 70% to 75%. The paste deterioration reveals a clear front of dissolution of Ca(OH)₂, which moves forward into the pastes with time, leaving behind a residual skeleton of partly decalcified C-S-H [38]. At ultimate stage of carbonation in cement paste, calcium carbonate, hydrated silica, alumina and ferric oxide are formed [47].
Another important aspect of carbonation is related to the corrosion of steel reinforcement. The protection of steel from corrosion by the alkaline condition of hydrated cement paste is neutralized by carbonation. The passive film on steel is destroyed at pH value between 9 and 10. If the entire concrete cover to steel is carbonated, corrosion of steel will occur where sufficient oxygen and water are available[47][51][52].

On the other hand, carbonation of concrete may have beneficial effect on the performance of concrete by increasing the compressive strength due to a dense superficial layer of calcite microcrystals [52]. In an experimental program aiming at evaluation of the diffusion rates of Cl, I and Cs through hardened cement paste, F. A. Sarott et al. [53] noticed that carbonation reaction significantly reduced the diffusion rates and hydraulic conductivities probably via the precipitation of CaCO₃ in the near surface pore space.

Atmospheric carbon dioxide can only react with the cement hydrates when there is sufficient pore water to dissolve it. Hydrated cement dried at a low relative humidity does not react to any significant extent. Although the carbonation reaction can occur readily under saturated conditions when CO₂ is freely available, in practice it is necessary for CO₂ to diffuse through the carbonated surface layer to reach the reaction zone. This gaseous diffusion is a slow process if the pores of the cement paste matrix are filled with water. The maximum rate of carbonation is observed at an intermediate moisture condition [54]. Under normal atmospheric condition, carbonation depth at a given time is approximately proportional to the paste porosity and inversely proportional to the square root of the total concentration of the free lime in concrete.
2.3.2 Accelerated carbonation test

The carbon dioxide content of normal atmosphere is 0.03% in rural air, 0.1% in unventilated laboratories and 0.3% in large cities[47]. To accelerate the carbonation process, the most commonly used method is to introduce high concentration of carbon dioxide gas to a test chamber under normal pressure, controlled humidity and controlled temperature [45][56][57][58]. The concrete specimens are placed in the test chamber for a selected period of time, e.g. 1, 7, 14, or 36 days and then removed for carbonation depth measurement or pH value analysis. The carbon dioxide content in the test chamber varies in from 10% to 100% and humidity from 40% to 90%, depending on researchers. As quoted by C. Levy [56], carbonation is optimum between 50 and 70% relative humidity. The acceleration of a test of this sort is equal to the ratio of the CO₂ concentration under the test condition to the CO₂ concentration under the normal atmosphere.

E. J. Reardon et al. proposed a method based on high pressure to accelerate the carbonation reaction in cementitious materials. Using this method, samples are placed in a stainless steel pressure-cell with an inner volume of 240 cm³ where they are exposed to carbon dioxide gas at various pressures up to 800 psi, which is near the liquefication pressure of carbon dioxide at ambient temperatures. Compressed gas cylinders of “Bone Dry” or “Analytical” grade carbon dioxide gas (99.5% purity) are used as pressure source. The uptake of CO₂ by samples is monitored by periodically weighing the sample cell maintained at a constant CO₂ pressure or measuring the pressure decrease with the cell sealed after the initial pressurization. High pressure carbonation is a potentially useful technique for studying the long-term physical effect of cementitious materials. It
significantly accelerates the carbonation reaction. For instance, at the pressure of 600 psi, the carbon dioxide uptake is noticeable after only 15 hours. However, the application of carbon dioxide at high pressure alters the nature of the carbonation process as compared to carbonation under normal atmosphere.

2.3.3 Determination of carbonation depth

Most authorities [51][54] agree that a measurement of the presence and extent of carbonation can be obtained by spraying a freshly fractured face of concrete with a dilute solution, usually in the range of 0.5% to 2%, of phenolphthalein in alcohol. The phenolphthalein indicator gradually changes from colorless to red as the pH rises from 8.3 to 10.0. The accuracy of this method is satisfactory for most surveys to determine the potential for carbonation induced corrosion. The procedure is simple, cheap and safe. The interpretation of results is easy. Other indicator can be used, but phenolphthalein provides the best color contrast between zones of high and low alkalinity. Such tests, however, only indicates the depth at which the pH is about 10.0, a pH likely to permit the onset of reinforcing steel corrosion. It does not necessarily correspond to either the boundary between uncarbonated and partially carbonated concrete or the boundary between partially carbonated and fully carbonated concrete.

L. Curtil et al. [60] introduced a more accurate method for evaluation of carbonation depth in concrete: the polarizing microscope method. During the microscopic examination, the light must pass through the samples. Therefore it is necessary to make a thin section of sample, which must be thirty microns thick. Study by polarizing microscope is composed of two parts. First an observation of the form, the colour, the break, the
cleavages of minerals is made under plane-polarized light. Then with cross polars, the birefringence and the extinction angle of minerals are examined. By locating the carbonation components in cement paste, particularly calcite and portlanite, mineral forms of CaCO₃ and Ca(OH)₂ respectively, the transition between carbonated zone and uncarbonated zone can be identified. The polarizing microscope gives clear pictures of the carbonation of concrete or mortar by identifying the mineralogical components and therefore gives an accurate measurement of carbonation depth.

Recent works [61][62][63] have also shown that stable isotope (¹³C, ¹⁸O) gives useful information on the carbonation problem of concrete. Using this method, the concrete sample powder is desiccated under vacuum then acidified through a large excess of 100% H₃PO₄ at 25°C in a calibrated volume. This is the classical technique for preparation of CO₂ from carbonate in highly reproducible conditions for isotope analysis. Isotope measurements on carbonate are presented in δ units, which represent the relative isotope concentration of a sample vs an international standard. The use of natural radiation of the ¹³C and ¹⁸O stable isotopes gives information on the origin and evolution of carbonate, using the fact that sources of material possesses distinct isotope. It also gives information on the processes of carbonation, as chemical reactions are generally accompanied by isotope fractionations. This technique is simple and low-cost.

A number of other alternative methods are available for monitoring carbonation, although none is as convenient as the phenolphthalein indicator method [51][54][61]. X-ray diffraction (XRD) can be used to measure the reduction of Ca(OH)₂ and the increase of CaCO₃. The amount of bound CO₂ can be determined by chemical extraction. Chemical
analysis can also provide an accurate pH value to indicate the occurrence of carbonation. Infra-red (IR) absorption can be used to monitor the formation of silica gel when calcium silicate hydrates carbonates. Differential thermal analysis (DTA) and thermal gravity (TG) test can be used to track the formation of CaCO₃.

2.4 Accelerated Artificial Weathering Test

Ultra violet ray usually produces little damage on concrete surface and is not a matter of concerns to researchers. Literature on this field is barely found. However, when organic coatings or sealants are applied to concrete structure, the weathering effect on the organic agents becomes significant.

In an extensive research program conducted by D. W. Pfeifer and M. J. Scali [3], two accelerated weathering test methods were used. One test method emphasizes alternate exposure of a coated concrete slab surface at 21°C to a solution of 15% NaCl salt water followed by exposure to ultra-violet light and infrared heat at 38°C with the salt water solution removed. This procedure simulates the alternate wet and dry environment found in southern climate coastal regions and is called southern climate test. The other method is called northern climate test. The test method is based on a daily cycles, with no activity on weekends. Each cycle consists of 15 hours of freezing at -9°C with no ponding, 2 hours of thawing at 21°C, 3 hours of ultraviolet radiation and infrared heat at 38°C and 3 hours of soaking with a 15% sodium chloride and 0.02 molar sulfurous acid water solution. Before refreezing, the surface is rinsed with tap water. Both tests require 24 week to complete. The performance of the tested slabs is judged by visual observations of the surface condition, by taking periodic copper-copper sulfate half cell corrosion potential
readings on the cracked concrete slabs and by measuring the chloride contents in the unreinforced concrete slabs at the end of the weathering tests.

William Cura [8] conducted another type of accelerated weathering test to evaluate the effectiveness of sealers for concrete bridge decks. In this method, sealed mortar coupons are exposed to accelerated weathering in an Atlas XWR Weather-Ometer on a cycle of 102 minutes of sunshine followed by 18 minutes of sun shine and rain. Two durations of exposure are used: 500 hours and 1000 hours. Upon completion of the weathering, the specimens are performance-tested via 28 days of saline immersion, in which the solution is cycled between 25°C and 60°C every 24 hours. At the end of the 28 days, the specimens are removed to 5% saline solution at room temperature for 4 hours to stabilize, rinsed, towel-dried to a surface saturated condition and weighed followed by a freeze/thaw cycling in a Scientemp Enviromental Chamber. The effect of 6 cycles per day for a total of 300 cycles is monitored by visual examination of the sealed surfaces.

In Hong Kong Specification for Civil Engineering Works [64], the quick ultra-violet artificial weathering test for concrete coatings is specified as 6 hours of UV-A light at 60°C ± 2°C followed by 6 hours of condensation at 50°C ± 2°C for a total of 4000 hours. At the end of the weathering cycles, the specimens are inspected visually for chalking, cracking, blistering, colour change and flaking.

2.5 Bond Strength between Concrete and Mortar

Literature search results in very limited findings related to the bond strength between concrete and cement mortar.
P. D. Stewart et. al. [4] examined the effect of coatings on the bond of bituminous overlays in terms of shear resistance of the bonds. 4” Marshall molds are used to form the concrete half of a test specimen. The concrete is vibrated to approximately half-depth in the mould and cured under sufficient moist condition for 4 days. After curing, the top surface of each specimen is rubbed on a wet concrete block to grind off any high points and surface laitance. The coating materials are then applied to the surface. Allowing sufficient curing time, a bituminous mixture is placed on the top of the coated concrete sections. The composite specimens are compacted with 100 blows of the Marshall hammer and stored for a minimum of one month before being tested. The bonding strength is measured by the amount of force required to shear the bituminous layer from the concrete. A loading apparatus is fabricated to ensure a shear force to be applied to the bond surface. With this method, it is found that the presence of linseed oil antispalling compound offered a very low bond strength between the treated concrete surface and bituminous overlay.

ASTM C952-86 [65] provides a typical method for evaluating the bond strength between mortar and masonry units. Based on this method, two concrete masonry units are placed crosswisely with their middle portions bonded together by a proper mortar to form a test couplet. After 28 days of curing, the two concrete units forming the couplet are pushed apart in a universal test machine, using a set of specially designed loading jigs. The bond strength is calculated based on the applied load and contact area between mortar and concrete.
CHAPTER 3. MATERIALS

All concrete specimens used in the tests were fabricated in Hong Kong. The concrete preservatives were provided by the Flax Council of Canada. This chapter gives the detailed information related to the concrete preservatives and the materials used for producing concrete specimens.

3.1 Concrete Preservatives

Four types of linseed oil-based concrete preservatives provided by the Flax Council of Canada and tested in this investigation are listed in Table 3-1.

Table 3-1. The Tested Concrete Preservatives

<table>
<thead>
<tr>
<th>Concrete Preservative</th>
<th>Batch Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type #P1</td>
<td>1019-D1004</td>
</tr>
<tr>
<td>Type #P2</td>
<td>1019-D2004</td>
</tr>
<tr>
<td>Type #P3</td>
<td>1019-D3004</td>
</tr>
<tr>
<td>Type #P4</td>
<td>1019-D4004</td>
</tr>
</tbody>
</table>

3.2 Cement, Aggregates and Admixture

The cement used for preparing concrete and mortar was Emerald Brand Portland cement supplied by China Cement Company (Hong Kong) Limited. Table 3-2 lists the technical data of the cement, including physical and chemical properties. The cement meets all requirements of BS 12:1991 for Portland Cement of strength class 52.2 N.

The aggregates used for preparing concrete mixes were supplied by the Pioneer Quarries (HK) Ltd. Table 3-3 lists the sieve analysis of the coarse and fine aggregates.
Table 3-2. Manufacturer’s Test Report for the Cement Used

<table>
<thead>
<tr>
<th>Property</th>
<th>Typical Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength at</td>
<td></td>
</tr>
<tr>
<td>2 days</td>
<td>22.7 MPa</td>
</tr>
<tr>
<td>7 days</td>
<td>40.7 MPa</td>
</tr>
<tr>
<td>28 days</td>
<td>58.9 MPa</td>
</tr>
<tr>
<td>Setting times,</td>
<td></td>
</tr>
<tr>
<td>Initial</td>
<td>80 minutes</td>
</tr>
<tr>
<td>Final</td>
<td>125 minutes</td>
</tr>
<tr>
<td>Soundness</td>
<td>0.1 mm</td>
</tr>
<tr>
<td>Additives</td>
<td>None</td>
</tr>
<tr>
<td>Chloride content</td>
<td>0.009 %</td>
</tr>
<tr>
<td>Fineness</td>
<td>335 m²/kg</td>
</tr>
<tr>
<td>Sulfate of cement as SO₃</td>
<td>2.5 %</td>
</tr>
<tr>
<td>Minor constituents</td>
<td>None</td>
</tr>
<tr>
<td>Chemical composition of clinker used</td>
<td></td>
</tr>
<tr>
<td>Silicon dioxide</td>
<td>22.0 %</td>
</tr>
<tr>
<td>Iron (III) oxide</td>
<td>3.3 %</td>
</tr>
<tr>
<td>Aluminum oxide</td>
<td>5.7 %</td>
</tr>
<tr>
<td>Calcium oxide</td>
<td>66.9 %</td>
</tr>
<tr>
<td>Magnesium oxide</td>
<td>1.0 %</td>
</tr>
<tr>
<td>Calcium silicate</td>
<td>78 %</td>
</tr>
<tr>
<td>Ratio, CaO/Silica</td>
<td>3.0</td>
</tr>
</tbody>
</table>

| Alkali content as sodium oxide equivalent on last 25 composite samples | Mean: 0.47; S. D. 0.08% |

Table 3-3. Typical Sieve Analysis of Aggregates (% Passing)

<table>
<thead>
<tr>
<th>B. S. Sieve</th>
<th>Granite-Coarse Aggregate, 20 mm Single-sized</th>
<th>Granite-Coarse Aggregate, 10 mm Single-sized</th>
<th>Granite-Crush Rock Fines, Grading Limits C</th>
</tr>
</thead>
<tbody>
<tr>
<td>37.5</td>
<td>100.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>95.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>50.0</td>
<td>100.0</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>8.8</td>
<td>92.5</td>
<td>100.0</td>
</tr>
<tr>
<td>5</td>
<td>1.0</td>
<td>16.5</td>
<td>97.9</td>
</tr>
<tr>
<td>2-36</td>
<td>2.5</td>
<td>76.0</td>
<td></td>
</tr>
<tr>
<td>1.18</td>
<td></td>
<td></td>
<td>53.5</td>
</tr>
<tr>
<td>600 μm</td>
<td></td>
<td></td>
<td>31.2</td>
</tr>
<tr>
<td>300</td>
<td></td>
<td></td>
<td>18.1</td>
</tr>
<tr>
<td>150</td>
<td></td>
<td></td>
<td>8.2</td>
</tr>
<tr>
<td>75</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pan</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

25
A retarding type water reducing / plasticising admixture for concrete, Daratard 17, was used to prepare the concrete mixes. The admixture was produced by Grace Construction Products.

### 3.3 Concrete Specimens

All concrete specimens were fabricated in Hong Kong. Two types of concrete mixes were used, as listed in Table 3-4. Concrete mix G 30/20 was specified by the Hong Kong Housing Authority and concrete mix G 45/20 was specified by the Hong Kong Highway Department. The concrete specimens were fabricated in three different sizes, 300 x 300 x 25 mm, 500 x 500 x 50 mm and 1500 x 500 x 50 mm. Specimens from concrete mix G 30/20 were cast on the site of the 80 of 1993 project -- Tin Shui Wai Area 13 Phase 2 N. T. Piling & Pilecaps to Foundation Piling, located in Hong Kong, using the same concrete mix as the project. The average 28-day compressive strength was 52 MPa. Specimens of concrete mix G 45/20 were cast on the site of TW/74/90 project -- Completion of Texco Rd./Tsan Wan Bypass Interchange & Improvement to Texco Rd. Phase 2, located in Hong Kong, using the same type of concrete mix as the project. The average 28-day compressive strength was 61 MPa. All concrete specimens were cured for at least 2 months before application of the preservatives.

<table>
<thead>
<tr>
<th>Mix, Slump</th>
<th>Cement</th>
<th>Aggregate</th>
<th>Free Water</th>
<th>Admixture ml/m³</th>
<th>A/C Ratio</th>
<th>W/C Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>30/20, 75 mm</td>
<td>360</td>
<td>655</td>
<td>505</td>
<td>620</td>
<td>185</td>
<td>720</td>
</tr>
<tr>
<td>45/20, 75 mm</td>
<td>470</td>
<td>670</td>
<td>515</td>
<td>500</td>
<td>185</td>
<td>940</td>
</tr>
</tbody>
</table>
3.4 Mortar

It was instructed that the mix design of the typical cement mortar used by Hong Kong Housing Authorities had not been controlled in terms of cement / sand ratio and water / cement ratio. Mortar is normally prepared for proper workability on site by contractors according to experience. The mortar for test of bond strength between concrete and mortar of this program was designed based on the same principle. The mix design was obtained through trial tests conducted at the University of Hong Kong and the mutual agreement between the University of Hong Kong and the University of Manitoba. The mortar was prepared at the two universities as the test proceeded, using the same types of cement and sand as the concrete. The water/cement ratio was equal to 0.45 and the cement/sand ratio was equal to 2.0.
CHAPTER 4. TEST PROCEDURES

The experimental program was conducted simultaneously at the University of Manitoba and the University of Hong Kong following a test manual [66]. This section provides a summarized description of the test procedures for application of preservatives, salt spray test, carbonation test, QUV weathering test, bond strength test and dripping test.

4.1 Application of Preservatives

A Graco spray machine was used to apply the preservatives. During application of the preservatives, the specimens of 300 x 300 x 25 mm and 500 x 50x x 50 mm were placed horizontally with the test surface oriented upward. The non-moulded surface was used as the test surface at the University of Hong Kong and the moulded surface was used as the test surface at the University of Manitoba. Each specimen was covered by one preservative. A total of eight 1500 x 500 x 50 mm specimens were used for the dripping test. Four of the eight specimens was placed vertically with the non-moulded surfaces as test surface at the University of Hong Kong and with the moulded surface as test surface at the University of Manitoba. The other four specimens were placed horizontally with moulded surface facing down as test surface at the University of Hong Kong and with the non-moulded surface facing down as test surface at the University of Manitoba. Each test surface was divided into two half portions with the dividing line parallel to the 1500-mm sides. The two half portions of each test surface were covered by two different preservatives respectively. Photographs in Figure 4-1 and Figure 4-2 show the application
site for the specimens at the University of Manitoba. Table 4-1 lists the sizes, quantities and test surface of the concrete specimens used by the two universities.

Before application of the preservatives, the concrete surfaces were cleaned using brushing followed by high pressure air blasting. The air for air blasting was oil and moisture free. The application of preservatives was performed under normal room conditions.

During the application, the 300 x 300 x 25 mm and 500 x 500 x 50 mm specimens were placed horizontally with the test surface facing up. The 1500 x 500 x 50 mm specimens for horizontal application were supported by shaw horses with their test surface facing down. The 1500 x 500 x 50 mm specimens for vertical application were placed vertically with their long sides perpendicular to the floor.
Figure 4-1. Application Site for the Vertical Specimens

Figure 4-2 Application Site for the Horizontal Specimens
Table 4-1. Test Surface Used by the Two Universities

<table>
<thead>
<tr>
<th>Concrete Specimen</th>
<th>Test Surface of Concrete Specimen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td>Number</td>
</tr>
<tr>
<td>500 x 500 x 50 mm</td>
<td>30</td>
</tr>
<tr>
<td>300 x 300 x 25 mm</td>
<td>10</td>
</tr>
<tr>
<td>1500 x 500 x 50 mm Vertical</td>
<td>4</td>
</tr>
<tr>
<td>1500 x 500 x 50 mm Horizontal</td>
<td>4</td>
</tr>
</tbody>
</table>

4.2 Curing and Cutting of the Test Specimens

After 2 days of curing under room conditions, the preserved specimens were moved to and stored in the curing room until the dates of testing or cutting. The curing room was maintained at 25 ± 2°C and 60 ± 5% relative humidity.

Within a minimum of 42 days of conditioning in the curing room after the application, 100 x 100 x 50 mm square blocks were cut from the control and preserved 500 x 500 x 50 mm specimens for carbonation and salt spray tests. 75 x 75 x 25 mm square blocks were cut from the control and preserved 300 x 300 x 25 mm specimens for QUV weathering test. 200 x 90 x 50 mm rectangular blocks were cut from the control and preserved 500 x 500 x 50 mm specimens for bond strength test. All the 100 x 100 x 50 mm, 75 x 75 x 25 mm and 200 x 90 x 50 mm blocks were cut under dry condition.

Within 3 days and 42 days of curing after the application, 2.5"-diameter cores were extracted from the 1500 x 500 x 50 mm specimens for dripping test, using wet process, since the dry coring machine is not suitable to specimens with steel reinforcement.
Due to the limited space available in the carbonation cabinet and salt spray cabinet, the carbonation and salt spray tests for specimens from the two concrete mixes were conducted one after another. The first sets of concrete blocks for salt spray, carbonation and QUV tests were placed to the equipment and the tests were started as soon as the fabrication of the blocks for the specific test was completed. The debonding tests were started immediately after all concrete blocks and cores were fabricated. Concrete blocks and cores which had not been subjected to any test were wrapped with polyethene to avoid further reaction between the preservatives and the surroundings until the tests started, which was necessary for maximizing the compatibility of the same tests started at different time.
4.3 Salt Spray Test

This test follows the Hong Kong Specification for Civil Engineering Works [44]. Salt spray test equipment, constructed by the University of Manitoba, consists of a stainless steel test chamber, salt water supply system, temperature control system and humidity control system [67], as shown in Figure 4-3. It is capable of providing 4 hours salt water spray at 40°C followed by 8 hours drying at 40°C and 30% R.H. for a total of 1000 hours. The artificial sea water was made up with 30 ± 5 g/liter sodium chloride in distilled water, correspondent to 3% by weight sodium chloride solution, complying with the method specified by BS 3900: Part F12 [68].

The salt spray test was conducted using 100 x 100 x 50 mm concrete blocks, which were cut from the 500 x 500 x 50 concrete panels. Three duplicated blocks were used for each preservative in addition to the three control blocks from each concrete mix for each test. The final results were calculated as an average of the results from the three duplicated specimens.

Before test, the specimens were coated with a protective epoxy coating on all surfaces except the tested surface. The concrete blocks were then placed to the test chamber and subjected to 4 hours of salt water spray followed by 8 hours drying period at 30% relative humidity for a total of 500 hours and 1000 hours, respectively. The salt spray was controlled at a flow rate of 2.5 GPM (Galons Per Minute). After every 12 days of operation, the artificial sea water in the water tank was replaced by a newly prepared one. The temperature in the test chamber was controlled at 40 ± 2°C. During the test, the concrete blocks in the test chamber were repositioned at the end of every 3 or 4 days of
operation. The position of each blocks were assigned randomly each time. Figure 4-4 shows a inside view of the salt spray chamber during operation. Prior to each salt spray test, another two preserved blocks for each preservative applied to each concrete mix were splitted into two halves to measure the penetration depth of the preservatives.

Upon completion of the salt spray cycling, the concrete specimens were removed from the test chamber, washed with distilled water and dried under the normal room
condition. Four holes were drilled on the test surface of each tested block, using a Bosh hammer drill with a 1\textquoteleft\textquoteleft-diameter or a 7/8\textquoteleft\textquoteleft-diameter solid carbide bit. As the drilling proceeded, the concrete filings were collected for each 5 mm depth increment. The collected filings were used as samples for chloride content analysis. The chloride content analysis, conforming to BS 1881: Part 124 [69], was performed by the Plains Innovative Laboratory Services, Saskatoon, Saskatchewan for the samples obtained at the University of Manitoba and by Castco Testing Centre Ltd., Hong Kong for the samples obtained at the University of Hong Kong.
Figure 4-4. Inside View of the Salt Spray Chamber
4.4 Carbonation Test

This test method is similar to the one used by C. Levy [56]. The test setup, constructed by the University of Manitoba, includes a carbonation test chamber, 100% carbon dioxide gas cylinder and two washing bottles connected to the CO₂ inlet and outlet of the carbonation chamber, as shown in Figure 4-5.

100 x 100 x 50 mm concrete blocks cut from the 500 x 500 x 50 mm concrete panels were used as test specimens. Three duplicated blocks were used for each preservative in addition to the three control specimens from each concrete mix. The final results were calculated based on the average of the results from the three duplicated blocks.

All of the 100 x 100 x 50 mm concrete blocks were coated with a protective epoxy coating on all surfaces except the tested surface before performing the carbonation test. The specimens were placed in an air tight test chamber containing over 95% CO₂ and 90% relative humidity for 18 days or 36 days. The test temperature was 25 ± 3°C. The CO₂ inside the test chamber was continuously replaced in a flow rate of 500 ml/minute. The air pressure in the chamber was slightly higher than the atmosphere air pressure due to the presence of the two washing bottles. Prior to each carbonation test, another two preserved concrete blocks for each preservative applied to each concrete mix, were split into two halves to measure the preservative penetration depth.

Upon completion of the test duration, the concrete specimens were removed from the test chamber and sealed in airtight enclosures until the dates of sampling. During sampling, four holes were drilled on the test surface of each tested block, using a Bosh
hammer drill with a 1" or 7/8" solid drill bit. As the drilling proceeded, concrete filings were collected for every 5 mm depth increment and were used for pH value analysis. The concrete blocks were then splitted into halves or quarters. 1% phenolphthalein solution was sprayed onto the newly splitted surfaces to expose the carbonation zone. The carbonation depths were evaluated based on the profiles of the visible carbonation zones.

The pH value analysis was performed by the Plains Innovative Laboratory Services, Saskatoon, Saskatchewan for the samples obtained at the University of Manitoba and by Materialab Ltd., Hong Kong for the samples obtained at the University of Hong Kong.
4.5 Bond Strength Test

This test followed ASTM C952-91 for evaluation of bond strength between mortar and masonry units. 200 x 90 x 50 mm concrete blocks were used for the bond strength test. Every two blocks were bonded together crosswisely with the test surfaces facing each other to form a test couplet. The mortar used was made up with water/cement ratio equal to 0.45 and cement/sand ratio equal to 2.0. Three duplicated couplets were made for each preservative in addition to the three control couplets from each concrete mix. All the test couplets were cured under the condition of 25°C and over 65% relative humidity for a total of 28 days. At the end of the curing age, the concrete couplets were pushed apart using a universal testing machine, as shown in Figure 4-6. The bond strength are calculated based on the area of the mortar-concrete interface and the force applied during debonding. The average value from the three duplicated couplets was used as a final result. Prior to the construction of the couplets, another two preserved concrete blocks for each preservative applied to each concrete mix, were split into two halves to measure the preservative penetration depth.
Figure 4-6. Bond Strength Test Setup
4.6 Quick Ultraviolet Artificial Weathering Test

This test was based on the equipment specified by ASTM G 53-84 [70] and the procedure outlined in the Hong Kong Specifications for Civil Engineering Works [64]. The QUV Artificial Weathering Tester was manufactured by the Q-Panel Company of the United States. Specific specimen holders were made at the University of Manitoba to facilitate the concrete specimens, as shown in Figure 4-7.

The weathering test included two stages. The first stage examined the effect of QUV weathering cycles on the visual appearance of the test specimens. The second stage examined the carbonation and salt spray resistances of the specimens after completion of various QUV weathering cycles. Concrete blocks of 75 x 75 x 25 mm from the 300 x 300 x 25 mm concrete panels were used for the QUV weathering test followed by the carbonation test. Concrete blocks of 75 x 75 x 50 mm from the 500 x 500 x 50 mm concrete panels were used for the QUV weathering test followed by the salt spray test. Prior to the first stage, another two preserved concrete blocks for each preservative applied to each concrete mix, were split into two halves to measure the preservative penetration depth.

During the first stage, all the required concrete blocks were placed to the QUV tester at once and subjected to 6 hours of ultra-violet ray exposure at 60°C followed by 6 hours of water condensation at 50°C for a total of 500, 1000, 2000 and 4000 hours, respectively. At the end of each test duration, a certain number of the specimens were removed from the tester for photographing and visual inspections for colour change, chalking, cracking, blistering and flaking. Two duplicated blocks for each preservative
Figure 4-7. The QUV Artificial Weathering Tester with Specimen Holders
and as control specimens were used for the visual inspections after 500, 1000 and 2000 hours of QUV test. One block for each preservative and as control specimen will be used for the visual inspections after 4000 hours of QUV test.

The visual inspections of the test surface were performed according to various ASTM specifications [71][72][73][74][75][76]. The degree of colour change was rated as 1, 1-2, 2, 2-3, 3, 3-4, 4, 4-5 and 5 on a scale of 5, with 1 representing a complete colour change and 5 representing no evidence of colour change. The effects of chalking, cracking and flaking were all recorded in five degrees rated No. 2, 4, 6, 8 and 10 on a scale of 10, in which 10 representing no evidence of chalking, cracking and flaking respectively. The effect of blistering was examined based on two characteristics of blistering: size and frequency. The size of blistering were evaluated on the basis of a numerical rating of 0 through 10 in four steps. With a 10 rating for no blister, sizes 8, 6, 4 and 2 were represented in increasing blister size. Correspondent to each blister size, four blister frequencies rated as Dense, Medium Dense, Medium, and Few were used.

There was an interval of at least 2 weeks between the first and second stages of the test to allow the concrete specimens after the required QUV weathering cycles to be conditioned at 25 ± 3°C and 60 ± 5% relative humidity.

During the second stage, the concrete specimens after 100, 200, 500, 1000, 2000 and 4000 hours of QUV weathering cycles were placed to the carbonation chamber for 18 days as described in Section 4.4. Subsequently, the specimens were split into two and sprayed with phenolphthalein for carbonation depth measurement.
Another set of specimens after 100 and 500 hours of QUV weathering cycles were subjected to a 500-hour salt spray test as described in Section 4.3. At the end of the salt spray test, the specimens were washed with distilled water to clear the salt precipitates on the test surface. The top 5 mm layer of each test surface was removed and discarded using a Bosh hammer drill with 1” or 7/8” solid drill bit. In the same hole with the same drill bit, a further 10 mm depth was achieved and the fillings was collected as a sample for chloride content analysis.
4.7 Dripping Test

The dripping test was conducted to examine the penetration ability of the preservatives upon concrete specimens of different finishes and in different orientation. 1500 x 500 x 50 mm concrete panel were used as the test specimens. The preservatives were applied to the test surface as described in Section 4.1. After 3 and 42 days following the application, 2.5"-diameter cores were extracted from the concrete specimens at selected locations as shown in Figure 4-8. A concrete disk of approximately 1" thick was cut from the preserved side of each core and was split into halves or quarters. Water was then sprayed onto the newly-split surfaces to delineate the preservative penetration zone. The penetration depths were measure based on the visible penetration zone profiles. Every penetration depth result was the average of ten readings taken along the penetration zone.
Figure 4-8 Locations of Cores for Dripping Test
CHAPTER 5. RESULTS AND DISCUSSION

This chapter provides a summary and discussion of all the test results. Discussions are mainly based on the results from the University of Manitoba, which are well in consistence with the results obtained by the University of Hong Kong. More detailed test data including colour photographs of the concrete specimens before and after each individual test and all the actual readings taken from each concrete specimen can be found in references [78] and [79]. A total of more than 500 concrete blocks or cores of various sizes were cut or extracted from the original concrete panels and used as specimens by each university for salt spray, carbonation, artificial weathering, bond strength or dripping test, in accordance with the test manual [66]. Data presented in this chapter are the average values of duplicated specimens in various tests.

5.1 Application of Preservatives

During the spray application, plastic sheets were placed under all the specimens and behind the vertical specimens to collect the splashing preservatives. The actual coverage rates of the preservatives on the concrete specimens were calculated based on data collected, before and after the spray application, from the weight of the plastic sheets, the styro-forms, and the preservative container feeding the spray gun. The spray gun and the long hose connecting the preservative container and the spray gun were not included in the calculation. The splashing loss beyond the range covered by the plastic sheets and the styro-forms and the loss on the timber were not collected and thus also not included in the calculation. During the application on the horizontal downward specimens and the vertical
specimens, such a loss might not be negligible. The test area of concrete specimens oriented downward was relatively small (1500 x 250 mm for each preservative). Due to the feature of this application, more splashing loss to the air could be expected. In addition, it was observed that certain amount of preservatives was squeezed into the gaps between the plastic sheets and the supporting shaw horses, which was also not included in the calculation. A similar situation was faced by the vertical specimens. As shown in Figure 4-1, 2'' x 4'' timber pieces were used to hold the styro-form during application of preservatives. The timbers were not included in the measurement of splashing losses, since the timber pieces were too heavy in comparison with the splashing losses of the preservatives. Its inclusion in the measurement would reduce the accuracy of the data. Therefore, among the measured coverage rates from the three specimen orientations, the coverage rates taken from the specimens oriented upward horizontally the most accurate.

Table 5-1 gives the results of the measured coverage rates and the number of spraying passes applied to the concrete specimens used in this program. Table 5-2 gives the data used to control the spray operation.

It was observed that dripping of the preservatives was significant for the vertical specimens immediately after the spray operation. No dripping was observed from the surface of the horizontal specimens oriented downward, however it was noticed that the preservatives form few drops in a scattered configuration on the surface.

Specimens coated by preservatives P1 and P2 dried at a similar rate. Most of the coated surface became completely dry within 18 hours after the application. Preservatives P1 and P2 on the rough (non-moulded) surfaces dried faster than those on the smooth
(moulded) surfaces. Some moulded surfaces coated by preservatives P1 and P2 remained tacky even after 3 days from the time of application. Specimens preserved by preservatives P3 and P4 dried at a faster rates. Complete dryness of P3 and P4 occurred within 4 and 7 hours, respectively, after the application. It should be noted that the dryness rates were related to the absorption rates of the preservatives. In terms of absorption rates, the four preservatives used in this investigation could be ranked in the following order: P4, P3, P1 and P2. Table 5-3 summarizes the observation related to the drying of the preservatives after application.

Table 5-1. The Actual Coverage Rate Measurement

<table>
<thead>
<tr>
<th>Preservative</th>
<th>Passes Applied</th>
<th>Specimen Orientation</th>
<th>Coverage Rate kg/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>4</td>
<td>Horizontal upward</td>
<td>0.152</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Horizontal downward</td>
<td>0.357</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Vertical</td>
<td>0.284</td>
</tr>
<tr>
<td>P2</td>
<td>3</td>
<td>Horizontal upward</td>
<td>0.145</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Horizontal downward</td>
<td>0.241</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Vertical</td>
<td>0.16</td>
</tr>
<tr>
<td>P3</td>
<td>3</td>
<td>Horizontal upward</td>
<td>0.107</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Horizontal downward</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Vertical</td>
<td>0.079</td>
</tr>
<tr>
<td>P4</td>
<td>3</td>
<td>Horizontal upward</td>
<td>0.127</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Horizontal downward</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Vertical</td>
<td>0.108</td>
</tr>
</tbody>
</table>
Table 5-2. Technical Data for the Spray Operation

- Air pressure into the spray unit: 60 - 65 psi
- Pressure at the tip of spray head: 2000 psi
- Temperature at the spray unit: 60 °C
- Temperature at the manifold: 35 - 40 °C
- Spray distance: 8 - 13 inches

Table 5-3. Drying and Absorption of the Preservatives on Concrete Surface

<table>
<thead>
<tr>
<th>Preservative</th>
<th>Concrete Surface</th>
<th>Drying Time</th>
<th>Rank of Absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>Moulded</td>
<td>most: &lt;18 hours</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>some: &gt;3 days</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Non-moulded</td>
<td>faster than the moulded one</td>
<td></td>
</tr>
<tr>
<td>P2</td>
<td>Moulded</td>
<td>most: 8 - 18 hours</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>some: &gt;3 days</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Non-moulded</td>
<td>faster than the moulded one</td>
<td></td>
</tr>
<tr>
<td>P3</td>
<td>Non-moulded &amp;</td>
<td>4 - 7 hours</td>
<td>2</td>
</tr>
<tr>
<td>P4</td>
<td>Moulded</td>
<td></td>
<td>1</td>
</tr>
</tbody>
</table>
5.2 Salt Spray Test

By visual inspection, it was found that after 500 and 1000 hours of testing, the originally smooth surfaces of the concrete blocks without preservative became obviously rough and those of the preserved blocks remained smooth. The brown colour of the concrete blocks was caused by the corrosion of some pipe fittings in the salt water supply system.

For concrete G30/20, the results of chloride content analysis are summarized in Figure 5-1 and Figure 5-2 with the data from Table 5-4. The results are categorized based on the nominal depths in concrete, which represent the depth increments for which the concrete filings were collected. For example, samples correspondent to nominal depth of 10 mm are the samples collected between the depths of 5 mm and 10 mm from the tested surface. The mean value is the average of the three individual values of the duplicated specimens for the given preservative. The results clearly indicate that all specimens coated by the concrete preservatives have very low chloride contents in comparison to the non-preserved concrete at any given depth level. Using any of the four concrete preservatives have significant effect on preventing chloride penetration into the concrete specimens. A chloride content ratio is used to evaluate the resistance of preserved concrete to chloride penetration. At the same concrete depth level, the ratio is defined as the ratio of the chloride content of the preserved concrete to the chloride content of the non-preserved concrete. A small chloride content ratio indicates a high resistance to chloride penetration. Figure 5-3 and Figure 5-4 show the comparison of the preservatives in terms of chloride content ratio. It can be found that the chloride content ratios of concrete preserved by P2
are all less than 0.1, indicating that preservative P2 provided the concrete surface with a resistance to chloride penetration ten times better than the non-preserved concrete surface. The resistance of the four preservatives to chloride penetration can be ranked as: P2, P1, P3 and P4.

Similarly, the results of chloride content analysis and resistance to chloride penetration for concrete G45/20 are given in Table 5-5 and Figure 5-5 and Figure 5-8. In protecting concrete from chloride penetration, preservatives P1 and P2 are significantly better than P3 and P4. The resistance of the four preservatives to chloride penetration can be ranked as: P2, P1, P4 and P3.

Table 5-4. Results of Chloride Content Analysis in Salt Spray Test (Concrete: G30/20)

<table>
<thead>
<tr>
<th>Nominal Depth in Concrete</th>
<th>Preservative</th>
<th>Chloride Content %</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 mm</td>
<td>None</td>
<td>1.36</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0.52</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.65</td>
</tr>
<tr>
<td>10 mm</td>
<td>None</td>
<td>0.93</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0.23</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.34</td>
</tr>
<tr>
<td>15 mm</td>
<td>None</td>
<td>0.63</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.07</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.17</td>
</tr>
</tbody>
</table>
Table 5-5. Results of Chloride Content Analysis in Salt Spray Test
(Concrete: G45/20)

<table>
<thead>
<tr>
<th>Nominal Depth in Concrete</th>
<th>Preservative</th>
<th>Chloride Content %</th>
<th>Duration of Salt Spray</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>500 hours</td>
<td>1000 hours</td>
</tr>
<tr>
<td>5 mm</td>
<td>None</td>
<td>1.78</td>
<td>1.86</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.26</td>
<td>0.32</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.18</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>1.64</td>
<td>1.67</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.85</td>
<td>0.92</td>
</tr>
<tr>
<td>10 mm</td>
<td>None</td>
<td>1.22</td>
<td>1.17</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.19</td>
<td>0.22</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.11</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0.44</td>
<td>0.53</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.41</td>
<td>0.30</td>
</tr>
<tr>
<td>15 mm</td>
<td>None</td>
<td>0.62</td>
<td>0.65</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>0.14</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.10</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0.24</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>0.13</td>
<td>0.17</td>
</tr>
</tbody>
</table>
Results of Chloride Content Analysis
(Concrete G30/20, 500 hours)

Figure 5-1. Results of Chloride Content Analysis for G 30/20 Concrete after 500 hours of Salt Spray Test

Results of Chloride Content Analysis
(Concrete G30/20, 1000 hours)

Figure 5-2. Results of Chloride Content Analysis for G 30/20 Concrete after 1000 hours of Salt Spray Test
Resistance to Chloride Penetration
(Concrete G30/20, 500 hours)

Figure 5-3. Resistance to Chloride Penetration for G 30/20 Concrete after 500 hours of Salt Spray Test

Resistance to Chloride Penetration
(Concrete G30/20, 1000 hours)

Figure 5-4. Resistance to Chloride Penetration for G 30/20 Concrete after 1000 hours of Salt Spray Test
Results of Chloride Content Analysis
(Concrete G45/20, 500 hours)

Figure 5-5. Results of Chloride Content Analysis
for G 45/20 Concrete after 500 hours of Salt Spray Test

Results of Chloride Content Analysis
(Concrete G45/20, 1000 hours)

Figure 5-6. Results of Chloride Content Analysis
for G 45/20 Concrete after 1000 hours of Salt Spray Test
Resistance to Chloride Penetration
(Concrete G45/20, 500 hours)

Figure 5-7. Resistance to Chloride Penetration for G 45/20 Concrete after 500 hours of Salt Spray Test

Resistance to Chloride Penetration
(Concrete G45/20, 1000 hours)

Figure 5-8. Resistance to Chloride Penetration for G 45/20 Concrete after 1000 hours of Salt Spray Test
5.3 Carbonation Test

The relation between the mean pH value and nominal depth in G30/20 concrete for preserved and non-preserved specimens are shown in Figure 5-9 and Figure 5-10 for the 18 and 36 days of carbonation respectively. Table 5-6 provides a summary of the pH value analysis results for G30/20 concrete specimens after 18 and 36 days of carbonation respectively, in which pH values are categorized based on the nominal sampling depths in concrete. The mean measured depth of carbonation for all specimens are plotted versus test duration in Figure 5-11 with the related data summarized in Table 5-7.

For the preserved and non-preserved specimens from G45/20 concrete, both pH value analysis and carbonation depth measurement showed no evidence of carbonation reaction. The pH values of all analyzed samples were well within the range of 12.4 to 12.7, which is the normal pH range of ordinary Portland cement. The carbonation depths of the specimens after 18 and 36 days of carbonation test were all too small to be measured and recorded as zero for mean values.

Although visual inspection of the surface indicates little difference between the concrete specimens before and after test, the pH value analysis and carbonation depth measurement show that the preservatives have significant effect on resisting carbonation of G30/20 concrete. The pH value of G30/20 concrete is around 12.60. When carbonation takes place, the most important effect is the reduction of pH value in concrete. From pH value analysis, it can be found that the pH values of the concrete specimens preserved by preservatives P1, P2 and P3 experienced little change during the 18 and 36 days of carbonation tests. However, the pH values of the unpreserved
specimens dropped dramatically in 18 days and even more in 36 days as shown in Figure 5-9 and Figure 5-10. These results indicate that carbonation reaction has penetrated at least 15 mm into the concrete. The concrete preserved by preservative P4 was an exceptional, in which the pH value after 36 days of test was even less than that of that of 18 days, showing an increase in carbonation resistance with the increase in test duration. The cause of such an unusual result is still unknown.

Carbonation depth measurement results confirm that preservatives P2 and P3 are very effective in improving the carbonation resistance of G30/20 concrete. Concrete specimens preserved by the two preservatives demonstrated zero carbonation depth after 18 and 36 days of tests. The average carbonation depths of concrete preserved by preservative P1 and P4 were less than half of the non-preserved ones. In consistence with the pH value analysis, the carbonation depth of concrete specimens preserved by preservatives P1 and P4 decreased as the test duration increased, indicating an increase in carbonation resistance.

The smooth side of both preserved and non-preserved G45/20 concrete specimens has high resistance to carbonation reaction. There is no basis to judge the effect of the concrete preservatives.
Table 5-6. Results of pH Value Analysis Test  
(Concrete: G30/20)

<table>
<thead>
<tr>
<th>Nominal Depth in Concrete</th>
<th>Preservative</th>
<th>pH Value</th>
<th>Duration of Carbonation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>18 days</td>
</tr>
<tr>
<td>5 mm</td>
<td>None</td>
<td>10.83</td>
<td>10.25</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>12.58</td>
<td>12.52</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>12.64</td>
<td>12.7</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>12.56</td>
<td>12.71</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>12.1</td>
<td>12.44</td>
</tr>
<tr>
<td>10 mm</td>
<td>None</td>
<td>11.01</td>
<td>10.37</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>12.59</td>
<td>12.53</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>12.61</td>
<td>12.69</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>12.57</td>
<td>12.7</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>12.35</td>
<td>12.43</td>
</tr>
<tr>
<td>15 mm</td>
<td>None</td>
<td>11.64</td>
<td>10.9</td>
</tr>
<tr>
<td></td>
<td>P1</td>
<td>12.61</td>
<td>12.53</td>
</tr>
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<td></td>
<td>P2</td>
<td>12.62</td>
<td>12.69</td>
</tr>
<tr>
<td></td>
<td>P3</td>
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<td>12.71</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>12.52</td>
<td>12.61</td>
</tr>
</tbody>
</table>

Table 5-7. Results of Carbonation Depth Measurement  
(Concrete: G30/20)

<table>
<thead>
<tr>
<th>Preservative</th>
<th>Carbonation Depth, mm</th>
<th>Duration of Carbonation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>18 days</td>
<td>36 days</td>
</tr>
<tr>
<td>None</td>
<td>21.08</td>
<td>27.77</td>
</tr>
<tr>
<td>P1</td>
<td>4.75</td>
<td>3.98</td>
</tr>
<tr>
<td>P2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>P3</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>P4</td>
<td>12.01</td>
<td>8.73</td>
</tr>
</tbody>
</table>
Results of pH Value Analysis
(Concrete G30/20, 18 days)

Figure 5-9. Results of pH Value Analysis for G 30/20 Concrete after 18 Days of Carbonation Test

Results of pH Value Analysis
(Concrete G30/20, 36 days)

Figure 5-10. Results of pH Value Analysis for G 30/20 Concrete after 36 Days of Carbonation Test
Results of Carbonation Depth Measurement (Concrete G30/20)

Figure 5-11. Results of Carbonation Depth Measurement for Concrete G 30/20
5.4 Bond Strength Test

The bond strength test results are summarized in Table 5-8. All the failures occurred on the interface between concrete and mortar. The results show that the presence of the concrete preservatives greatly reduces the bond between the preserved concrete surface and the mortar bed. Half of the concrete couplets failed before even being loaded, i.e. by supporting only the self weight of one 200 x 90 x 50 mm block, corresponding to about 0.004 MPa of strength. The highest bond strength between preserved concrete surface and mortar was provided by the preservative P1 on concrete G30/20, which was only about 10% of the bond strength between non-preserved concrete and mortar.

<table>
<thead>
<tr>
<th>Preservative</th>
<th>Bond Strength, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Type of Concrete</td>
</tr>
<tr>
<td></td>
<td>G 30/20</td>
</tr>
<tr>
<td>None</td>
<td>0.256</td>
</tr>
<tr>
<td>P1</td>
<td>0.032</td>
</tr>
<tr>
<td>P2</td>
<td>0.01</td>
</tr>
<tr>
<td>P3</td>
<td>0</td>
</tr>
<tr>
<td>P4</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 5-8. Results of Bond Strength Test
5.5 Quick Ultraviolet Artificial Weathering Test

The visual inspection results for colour change and chalking are given Figure 5-12 to Figure 5-15 and the related data are summarized in Table 5-9 and Table 5-10. Table 5-11 and Table 5-12 summarize the visual inspection results for cracking and blistering respectively. Visual inspection showed that there was no evidence of flaking on all tested concrete blocks. Each data presented in Table 5-9 and Table 5-10 is an average result from two duplicated specimens. For calculation purposes, the degree No. 1-2, 2-3 and 4-5 from the visual inspections are taken as 1.5, 2.5 and 4.5 respectively.

The results indicate that colours of all the preserved concrete surfaces were significantly changed after exposition to the ultra-violet ray. Longer exposition introduced greater colour change. The UV exposure had little effect on the colour of concrete surfaces without preservative, as shown in Figure 5-12 and Figure 5-13. Chalking of most of the preserved concrete specimens became greater than the non-preserved ones after 1000 hours of QUV test, as shown in Figure 5-14 and Figure 5-15. As shown in Table 5-11, the QUV exposure had little effect on the cracking of all test specimens. The non-preserved specimens and the specimens preserved by P3 and P4 showed no evidence of blistering. With the exception of concrete G30/20 preserved by P1, blistering occurred only occasionally on the specimens preserved by P1 and P2, as shown in Table 5-12.

The results of 18-day carbonation depth measurement after various duration of QUV test are shown in Table 5-13 and Figure 5-16. For comparison, the 18-day carbonation depth results from Table 5-7 are also included as the results correspondent to 0 hour of QUV exposure. It can be seen that the QUV artificial weathering cycles have
significant effect on the carbonation resistance of the preserved G30/20 concrete specimens. In general, the carbonation depths of all preserved concrete specimens increase as the QUV test duration. After 500 hours of QUV test, carbonation depths of concrete specimens preserved by all four preservatives are close or exceed the carbonation depth of the correspondent non-preserved concrete. The artificial weathering cycles seem destroying the penetrating preservatives and providing more passages for CO₂ molecules. The time limit for the preservatives to remained effective under the test conditions is not more than 500 hours. Increase of QUV durations has little effect on the carbonation resistance of non-preserved concrete. However, when compared with the specimens exposed to QUV weathering, the non-preserved specimen without subjected to QUV test have inferior resistance to carbonation.

The chloride content results of 500-hour salt spray test after 100 and 500 hours of QUV test are given in Table 5-14, Figure 5-17 and Figure 5-18, where notation “n/a” implies data not available due to limited number of specimens. For comparison, the chloride content results from the specimens after 500 hours of spray without QUV exposure are also listed in Table 5-14. Each 0-hour QUV result in Table 5-14 is an average of the measured results corresponding to nominal depths of 10 mm and 15 mm in Table 5-4 and Table 5-5., since the concrete samples for chloride analysis in salt spray test following QUV test were taken from the 75 x 75 x 50 mm concrete blocks within a 10 mm depth increment after removal of the top 5 mm layer, while the samples in single salt spray test were taken from 100 x 100 x 50 mm concrete blocks in every 5 mm depth increment.
Chloride contents from all the preserved concrete specimens are well less than the non-preserved ones. The four concrete preservatives greatly improve the resistance of concrete to chloride penetration even after exposing to QUV weathering cycles. The chloride content ratios of preserved concrete to non-preserved concrete range from 14% to 50% for concrete G30/20 and 23% to 85% for concrete G45/20, as shown in Figure 5-19 and Figure 5-20. Preservative P1 seems to be the most effective one. It is noticed that the chloride content ratio of 14% achieved by P1 on G30/20 concrete is almost the same for all QUV durations including the one without being subjected to QUV test, indicating that the QUV test has little effect on this preservative in terms of salt spray resistance.

From Figure 5-17 and Figure 5-18, it can also be found that the salt spray resistance of non-preserved concrete consistently increases with the increase of QUV test duration, which could be attributed to the saturated humidity and elevated temperature conditions inside the QUV tester benifiting the curing of concrete. The performance of preserved concrete specimens varies depending on concrete and QUV test duration.
### Table 5-9. Results of Visual Inspection for Colour Change

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Preservative</th>
<th>Test Duration, hours</th>
<th>500</th>
<th>1000</th>
<th>2000</th>
<th>4000</th>
</tr>
</thead>
<tbody>
<tr>
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<td>5</td>
<td>5</td>
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<tr>
<td></td>
<td>P2</td>
<td></td>
<td>4</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td></td>
<td>4.25</td>
<td>1.75</td>
<td>1.25</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td></td>
<td>2.5</td>
<td>2.5</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>G 45/20</td>
<td>None</td>
<td></td>
<td>5</td>
<td>5</td>
<td>5</td>
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<td></td>
<td>P1</td>
<td></td>
<td>1.5</td>
<td>1</td>
<td>1.25</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>P2</td>
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<td>2.75</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td></td>
<td>2.75</td>
<td>2</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td></td>
<td>2.5</td>
<td>2.5</td>
<td>1.5</td>
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</tbody>
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### Table 5-10. Results of Visual Inspection for Chalking

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<th>1000</th>
<th>2000</th>
<th>4000</th>
</tr>
</thead>
<tbody>
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<td>8</td>
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</tr>
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<td>6</td>
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</tr>
<tr>
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<td>6</td>
<td>6</td>
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<td></td>
<td>8</td>
<td>7</td>
<td>7</td>
<td>6</td>
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<td></td>
<td>6</td>
<td>6</td>
<td>5</td>
<td>6</td>
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<tr>
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<td>6</td>
<td>6</td>
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</tr>
<tr>
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<td>4</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td></td>
<td>8</td>
<td>8</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>P4</td>
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<td>6</td>
</tr>
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<td>Concrete</td>
<td>Preservative</td>
<td>Test Duration, hours</td>
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</tr>
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<td>--------------</td>
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<tr>
<td></td>
<td></td>
<td>500</td>
<td>1000</td>
<td>2000</td>
<td>4000</td>
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</tr>
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<td>10</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
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<td>10</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td></td>
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<td>10</td>
<td>10</td>
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<td></td>
</tr>
<tr>
<td></td>
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<td>10</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
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<td>P4</td>
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<td>10</td>
<td>10</td>
<td></td>
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<tr>
<td></td>
<td>P2</td>
<td>8</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td></td>
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<td>10</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
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Table 5-12. Results of Visual Inspection for Blistering

<table>
<thead>
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<th>Duplicated Specimens</th>
<th>Test Duration, hours</th>
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<tbody>
<tr>
<td></td>
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<td>G30/20</td>
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</tr>
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<td>1</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>P1</td>
<td>1</td>
<td>6, Dense</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>6, Few</td>
</tr>
<tr>
<td>P2</td>
<td>1</td>
<td>6, Dense</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>6, Few</td>
</tr>
<tr>
<td>P3</td>
<td>1</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>P4</td>
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<td>G45/20</td>
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<td>10</td>
</tr>
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<td></td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>P1</td>
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<td>10</td>
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<td></td>
<td>2</td>
<td>10</td>
</tr>
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</tr>
<tr>
<td></td>
<td>2</td>
<td>6, Dense</td>
</tr>
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<td>10</td>
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<tr>
<td></td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>P4</td>
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Table 5-13. Results of Carbonation Depth Measurement after QUV Test
(Concrete: G30/20, Carbonation Duration: 18 days)

<table>
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<th>Duration of QUV Test</th>
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<th></th>
</tr>
</thead>
<tbody>
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<td>None</td>
<td>P1</td>
<td>P2</td>
<td>P3</td>
<td>P4</td>
</tr>
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<td>21.08</td>
<td>4.75</td>
<td>0</td>
<td>0</td>
<td>12.01</td>
</tr>
<tr>
<td>100 hours</td>
<td>11.93</td>
<td>4.43</td>
<td>5.29</td>
<td>0</td>
<td>5.25</td>
</tr>
<tr>
<td>200 hours</td>
<td>17.22</td>
<td>2.44</td>
<td>6.18</td>
<td>4.93</td>
<td>8.45</td>
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<tr>
<td>500 hours</td>
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<td>13.25</td>
<td>10.74</td>
<td>12.52</td>
<td>31.22</td>
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<tr>
<td>1000 hours</td>
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<td>19.67</td>
<td>19.63</td>
<td>17.63</td>
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<td>17.64</td>
<td>18.22</td>
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<tr>
<td>4000 hours</td>
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<td>30</td>
<td>30</td>
<td>30</td>
<td>30</td>
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Table 5-14. Results of Chloride Content Analysis after QUV and Salt Spray Tests (%)
(Salt Spray Duration: 500 hours)

<table>
<thead>
<tr>
<th>Duration of QUV Test</th>
<th>Preservative</th>
<th>Concrete</th>
<th>G30/20</th>
<th>G45/20</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>None</td>
<td>P1</td>
<td>P2</td>
<td>P3</td>
</tr>
<tr>
<td>0 hour</td>
<td>0.78</td>
<td>0.10</td>
<td>0.05</td>
<td>0.18</td>
</tr>
<tr>
<td>100 hours</td>
<td>0.70</td>
<td>0.09</td>
<td>0.10</td>
<td>0.15</td>
</tr>
<tr>
<td>500 hours</td>
<td>0.48</td>
<td>0.07</td>
<td>0.16</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Concrete G30/20

|                      | None         | P1       | P2     | P3     | P4     |
| 0 hour               | 0.90         | 0.13     | 0.09   | 0.36   | 0.55   |
| 100 hours            | 0.35         | n/a      | 0.24   | 0.08   | 0.08   |
| 500 hours            | 0.30         | 0.10     | 0.11   | 0.21   | 0.26   |
Figure 5-12. Degree of Colour Change after QUV Test for Concrete G 30/20

Figure 5-13. Degree of Colour Change after QUV Test for Concrete G 45/20
Figure 5-14. Degree of Chalking after QUV Test for Concrete G 30/20

Figure 5-15. Degree of Chalking after QUV Test for Concrete G 45/20
Carbonation Depth Measurement After QUV Test

(Concrete: G30/20, Carbonation Duration: 18 days)

Figure 5-16. 18-day Carbonation Depth after QUV Test
Figure 5-17. Chloride Content for G30/20 Concrete after QUV and Salt Spray Tests

Figure 5-18. Chloride Content for G45/20 Concrete after QUV and Salt Spray Tests
Resistance to Chloride Penetration
(Concrete: G30/20, Salt Spray: 500 hours)

Figure 5-19. Chloride Content Ratio for G30/20 Concrete after QUV and Salt Spray Tests

Resistance to Chloride Penetration
(Concrete: G45/20, Salt Spray: 500 hours)

Figure 5-20. Chloride Content Ratio for G45/20 Concrete after QUV and Salt Spray Tests
5.6 Dripping Test

The test results for the dripping test are summarized in Table 5-15 to Table 5-17 and Figure 5-21 to Figure 5-25. The note "N/A" implies that the penetration depth of the specific specimen could not be identified, which might be either too large or too small. A figure summary of the 28-day dripping test results for vertical G45/20 concrete specimens is not provided due to the feature of the data as shown in Table 5-17.

For the horizontal downward specimens, the preservative penetration depth demonstrates little change with time. Concrete G45/20 seems easier than concrete G30/20 for the preservative to penetrate. The penetration ability of the preservatives P1, P3 and P4 are superior to preservative P2.

In vertical application, the penetration depth increases as the location of cores changes from top to bottom along the 1500 x 500 x 50 mm specimens. Both concrete G30/20 and G45/20 behave similarly in terms of penetration of the preservatives. The penetration depth of preservatives P1 and P2 increases with time while the penetration depth of preservatives P3 and P4 is difficult to be judged since the penetration front boundaries in most of the cores from the specimens for the 28-day test were not clear.

Penetration depth measurement was also performed using the concrete blocks for salt spray, carbonation, bond strength and QUV tests. The concrete specimens for these tests were oriented horizontally and facing upward when the preservatives were applied. Due to gravity effect which may help full penetration of the preservatives into concrete, it was not easy to identify the penetration front boundaries and consequently hard to measure the actual penetration depth. For vertical specimens and horizontal specimens
facing downward, the gravity is against the penetration of the preservatives. Consequently, the preservatives are more concentrated near the preserved surface and form visible penetration front boundaries.

Considering also the observations obtained in section 5.1, the penetration ability of the four preservatives can be ranked as P4, P3, P1 and P2. Concrete G45/20 seems easier for the preservatives to penetrate.

Table 5-15. Results of 3-day Dripping Test for Horizontal Specimens

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Preservative</th>
<th>Penetration Depth, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Curing Age, days</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3 days</td>
</tr>
<tr>
<td>G 30/20</td>
<td>P1</td>
<td>2.15</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>1.22</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>1.82</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>2.29</td>
</tr>
<tr>
<td>G 45/20</td>
<td>P1</td>
<td>3.57</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>1.7</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>2.53</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>2.45</td>
</tr>
</tbody>
</table>

Table 5-16. Results of 3-day Dripping Test for Vertical Specimens

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Preservative</th>
<th>Location of Coring (m)</th>
<th>Penetration Depth, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Top (1.4)</td>
<td>Mid-height (0.75)</td>
</tr>
<tr>
<td>G 30/20</td>
<td>P1</td>
<td>0</td>
<td>0.63</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0</td>
<td>0.63</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>1.23</td>
<td>1.78</td>
</tr>
<tr>
<td>G 45/20</td>
<td>P1</td>
<td>0</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.41</td>
<td>1.79</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0</td>
<td>1.03</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>1.55</td>
<td>1.78</td>
</tr>
</tbody>
</table>
Table 5-17. Results of 28-day Dripping Test for Vertical Specimens

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Preservative</th>
<th>Penetration Depth, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Location of Cores (height, m)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Top (1.4)</td>
</tr>
<tr>
<td>G 30/20</td>
<td>P1</td>
<td>0.92</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>0.83</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>1.65</td>
</tr>
<tr>
<td>G 45/20</td>
<td>P1</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>P2</td>
<td>1.58</td>
</tr>
<tr>
<td></td>
<td>P3</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>P4</td>
<td>N/A</td>
</tr>
</tbody>
</table>
Figure 5-21. Dripping Test Results for Horizontal Downward Specimens from Concrete G 30/20

Figure 5-22. Dripping Test Results for Horizontal Downward Specimens from Concrete G 45/20
Dripping on Vertical Specimens
(Concrete G30/20, 3 days)

![Graph showing dripping test results on vertical specimens from Concrete G30/20 within 3 days after the application.]

Figure 5-23. Dripping Test Results on Vertical Specimens From Concrete G 30/20 within 3 days after the Application

Dripping on Vertical Specimens
(Concrete G30/20, 28 days)

![Graph showing dripping test results on vertical specimens from Concrete G30/20 within 28 days after the application.]

Figure 5-24. Dripping Test Results on Vertical Specimens from Concrete G 30/20 within 28 days after the Application
Dripping on Vertical Specimens
(Concrete G45/20, 3 days)

Figure 5-25. Dripping Test Results on Vertical Specimens from Concrete G 45/20 within 3 days after the Application
CHAPTER 6. CONCLUSIONS

1. All four concrete preservatives P1, P2, P3 and P4 have significant effect on enhancing the resistance of concrete G30/20 and G45/20 to salt spray attack. Their effectiveness can be ranked in the order of P2, P1, P3 and P4. Concrete preserved by preservative P2 is nearly ten times better than non-preserved concrete in resisting chloride penetration.

2. The four concrete preservatives have significant effect on enhancing the resistance of G30/20 concrete to carbonation. Their effectiveness can be ranked in the order of P2, P3, P1 and P4. Preservatives P2 and P3 are equally effective. G30/20 concrete preserved by preservatives P2 or P3 has a carbonation depth of zero after 36 days of test in contrast to an average of more than 20 mm in non-preserved concrete. G45/30 concrete specimens have high resistance against carbonation even without preservatives. Therefore, no conclusion could be made regarding the effect of the four preservatives on improving the carbonation resistance of G45/20 concrete.

3. All the preserved concrete surfaces change colours after quick ultra violet ray artificial weathering test. After 1000 hours of weathering, UV ray introduces greater chalking on most of the preserved concrete surfaces in comparison to the non-preserved surface. However, it has little effect on the cracking, blistering and flaking of the preserved and non-preserved concrete surfaces.

4. QUV weathering has little effect on the carbonation resistance of non-preserved concrete but reduces the carbonation resistance of all preserved concrete specimens.

82
The duration limit of QUV weathering for the preserved concrete to remain superior to the non-preserved concrete is less than 500 hours.

6. QUV weathering test improve the salt spray resistance of non-preserved concrete. It has little effect on salt spray resistance of G30/20 concrete preserved by P1. After up to 500 hours of QUV weathering, all preserved concrete specimens demonstrate higher salt spray resistance than non-preserved ones, with the chloride content ratio to non-preserved concrete ranging from 14% to 50% for concrete G30/20 and 23% to 85% for concrete G45/20.

7. The preservatives reduce the bond strength between concrete and mortar. The bond strength between a preserved concrete surface and mortar is less than 10 percent of the bond between a non-preserved concrete surface and mortar.

8. All the four concrete preservatives are capable of penetrating through vertical, horizontally upward and horizontally downward concrete surfaces. The measurable penetration depths ranges from 0 to 3.57 mm. The penetration ability could be ranked for the four preservatives in the following order: P4, P3, P1 and P2. Concrete G45/20 appears easier than G30/20 concrete for the preservatives to penetrate.
CHAPTER 7. RECOMMENDATIONS FOR FUTURE WORKS

Preservatives P1 and P2 are recommended to the field test in the second phase of the program. The field test will be conducted in Hong Kong area only. To avoid the interference of existing concrete deterioration to the test results, the test sites should be selected at the recently completed concrete constructions. Three test sites will be needed.

Site 1  Concrete Columns:

Three concrete columns will be selected, of which each column should have at least one face exposed to open air, rain and direct sunlight. Two of the three columns will be coated with concrete preservatives P1 and P2 respectively. The third column will be used to extract control specimen.

Site 2  Concrete Walls

A concrete wall with at least one of its surfaces exposed to open air, rain and direct sunlight will be selected. The test site will be divided into three equal section. The size of each section will depend on the size of the selected site. It is suggested that each section have an dimension of 3 x 10 m to facilitate the spray operation. Two of the three sections will be coated by preservatives P1 and P2. The third section will be used to extract control specimens.

Site 3  Highways

For horizontal application, a concrete pavement or bridge deck will be selected. The concrete surface should be exposed to open air, rain and direct sunlight. The
test site will be divided into three equal sections. The size of each section will depend on the size of the selected site. It is suggested that each section have an dimension of $3 \times 10$ m to facilitate the spray operation. Two of the three sections will be coated by preservatives P1 and P2. The third section will be used to extract control specimens.

Four set of concrete cores will be extracted from the test sites within seven days, 6 months, 1 year and 2 years after application of the preservatives, using a Bosh hammer drill and a 2’’ core bit. Each set of cores contained 7 cores for each preservative and control samples from each test site, with one core of each for penetration depth measurement, carbonation depth measurement and chloride content analysis. 2 cores of each for salt spray test and carbonation test. The carbonation depth measurement and chloride content analysis are performed to evaluate the resistance of preserved and non-preserved field concrete to carbonation and chloride intrusion in natural atmosphere. The accelerated salt spray test and carbonation test are performed to evaluate the durability of the two concrete preservatives upon natural weathering. All the tests to be conducted using the cores extracted 7 days after the application will act as controls in the time scale.
CHAPTER 8. REFERENCE


86


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