# Effect of carbonation curing on efflorescence formation in concrete pavers

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## Abstract

Efflorescence is a salt deposit which is formed on or near the surface of a porous material such as Portland cement concrete. Although efflorescence is not an indication of internal damage, it alters the aesthetic quality of the product. The primary goal of this work was to investigate whether early-age carbonation can help reduce, or altogether eliminate, efflorescence formation on concrete paver blocks. The devised carbonation technique involved curing concrete pavers in a chamber filled with either pure or 20% carbon dioxide gas under a pressure of 5 bar. The carbonated pavers were evaluated by a newly developed test method to visualize efflorescence formation. It was modified from wicking test and was proven effective for the accelerated formation of efflorescence. A MATLAB-assisted image-analysis technique was used to quantify efflorescence severity. It was found that pure CO<sub>2</sub> curing seemed to be successful in eliminating the occurrence of efflorescence compared to the conventional hydration cured control specimens. Results from X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) coupled with Energy-dispersive X-ray (EDX) spectroscopy identified efflorescence salt deposits as a crystalline potassium sulfate. Carbonation curing was able to reduce the efflorescence potential in two different ways. First, the curing process consumed the calcium hydroxide, the necessary chemical component for carbonate- based efflorescence. Second, the formation of efflorescence requires water to migrate up to the surface. Carbonation curing technique could densify the concrete surface and decrease the absorption consequently. Moreover, compressive strength results showed carbonation's advantage in achieving rapid strength gain. The carbonation process could reduce the production cycle, facilitate carbon storage in concrete and eliminate the efflorescence formation on the surface.

## Résumé

L'efflorescence est un dépôt de sel qui s'est formée sur ou près de la surface du matériau poreux, tel que du béton de ciment Portland. Bien que l'efflorescence ne constitue pas une indication des dommages internes, il altère la qualité esthétique du produit. L'objectif principal de cet ouvrage était d'étudier si la première carbonatation peut aider à réduire ou éliminer complètement, la formation de l'efflorescence sur les blocs pavés de béton. La technique planifiée de la carbonatation a impliqué la cure des pavés en béton dans une chambre remplie de gaz dioxyde de carbone (soit pur, soit 20%) sous une pression de 5 bars. Les pavés carbonatés ont été évalués par une méthode de test qui est nouvellement développée afin de visualiser la formation de l'efflorescence. Cette méthode a été modifiée du test de mèche, et elle a été prouvée efficace pour la formation accélérée de l'efflorescence. Une technique d'analyse d'image assistée par MATLAB a été utilisée pour quantifier la sévérité de l'efflorescence. On a découvert que, par rapport à la cure de l'hydratation conventionnelle de contrôle des échantillons, la cure du CO<sub>2</sub> pur semble être efficace pour éliminer l'occurrence de l'efflorescence. Les résultats de diffraction des rayons X et de microscope électronique à balayage couplée à la spectroscopie des rayons X de l'énergiedispersive ont identifié les dépôts de sel de l'efflorescence comme le sulfate de potassium cristallin. La cure de carbonatation était capable de réduire le potentiel de l'efflorescence en deux manières différentes. Premièrement, le processus de cure a consommé l'hydroxyde de calcium, qui est le composant chimique nécessaire à l'efflorescence pour la base carbonatée. Deuxièmement, la formation de l'efflorescence a besoin de l'eau pour migrer vers la surface. Par conséquent, la technique de la cure de carbonatation peut densifier la surface du béton et diminuer l'absorption. De plus, les résultats de la résistance à la compression ont montré l'avantage de la carbonatation dans la réalisation de l'obtention rapide de la résistance. Le processus de carbonatation peut réduire

le cycle de production, faciliter le stockage de carbone dans le béton ainsi d'éliminer la formation de l'efflorescence sur la surface.

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# List of abbreviation

- ASTM: American Society for Testing and Materials
- CSA: Canadian Standards Association
- OPC: Ordinary Portland Cement
- SEM: Scanning Electron Microscope
- XRD: X-Ray Diffraction
- EDS: Electron Dispersive Spectroscopy

W/C ratio: Water-to-cement ratio (ratio of mass of water over mass of cement powder in cement paste)

### **<u>Cement chemistry notations</u>**

- C: CaO Calcium oxide or lime
- $\overline{C}$ : CO<sub>2</sub> Carbon dioxide
- H:  $H_2O$  Water
- S:  $SiO_2$  Silicon dioxide or silica
- A:  $Al_2O_3$  Aluminium oxide

- F:  $Fe_2O_3$  Iron oxide
- $C_3S: \qquad 3 \ CaO \cdot SiO_2 \text{ } Tricalcium silicate}$
- $C_2S:$  2 CaO · SiO<sub>2</sub> Dicalcium silicate
- $C_3A$ : 3 CaO · Al<sub>2</sub>O<sub>3</sub> Tricalcium aluminate
- CSH: Calcium silicate hydrates of various C/S ratio

# **Chapter 1 Introduction**

#### **1.1. General Overview**

Efflorescence is generally defined as the formation of salts deposits, usually white, occurs on or near the surface of Portland cement concrete and masonry units, including bricks and tiles. This can be a persistent problem, especially for darker colored products as a result of emphasized contrast. Normally, efflorescence is not an indication of internal damage, but mainly affects the aesthetic quality of the product (Adam 2002, Dow and Glasser 2003, Cement Concrete & Aggregates Australia 2006). Water migrating through the concrete carries dissolved salts or calcium hydroxide (CH) up to the surface of the product. Then efflorescence gradually appears either because the salts deposits after subsequent evaporation or CH reacts with CO<sub>2</sub> in the atmosphere forming calcium carbonate (Kompatscher, Ingrisch et al. 2007). Generally, efflorescence will not affect the mechanical properties and durability of the concrete. But it will create an undesirable appearance, likely incurring economic loss from products rejection (Norsuzailina, Sinin et al. 2014).

In principle, the presence of three essential components is necessitated for efflorescence formation: CH or dissolvable salts, water as transport media and pathways for water migration (Kompatscher, Ingrisch et al. 2007). Efflorescence can be eliminated by avoiding any of them. Early age carbonation curing, an alternative curing method using carbon dioxide to cure the concrete, seems as a likely candidate to mitigate efflorescence. Carbonation consumes the chemical component (CH) responsible for the carbonate-based efflorescence by obstructing the mechanism mentioned above. It also aids in the decreases of concrete's absorption, consequently reducing water permeation towards the service surface. In addition, several other benefits of this technique have been reported including fast strength gain, enhanced long-term durability and permanent carbon dioxide sequestration (Shao and Lin 2011).

Ducon Industries FZCO is a concrete paver manufacturer located in Dubai, founded in 1938. The company provides a wide range of design options, styles and colors of pavers and interlocks to suit different architectural styles and geographic locale. One of the common on-site challenges faced with colored pavers, especially dark-pigmented ones, is the formation of persistent discoloring efflorescence months after installation. For this reason, the aim of this study was to investigate the potentially-beneficial effect of early-age carbonation curing on service-life efflorescence of paver blocks. Ducon Industries' popular 8 x 8 x 6 cm cube paving block was used in this study (Figure 1-1). This product comes in an array colors, but in order to emphasize the color contrast of effloresced bocks, the black paver was chosen.



Figure 1-1: Ducon Industries Cube Paving Block with available colors

#### **1.2. Objectives**

This research attempted to reduce the efflorescence potential of concrete pavers via early age carbonation curing. A real commercial paver product from Ducon Industries was mimicked and it was used as the testing specimen throughout this study. The objectives of this research are:

- To develop the suitable efflorescence-inducing test method for concrete pavers. Secondary
  efflorescence normally takes months to form, and there are no ASTM or CSA standardized
  protocols for testing efflorescence of concrete (current standard is only for clay and bricks).
  Therefore, the three efflorescence testing methods are evaluated, including the wicking test,
  puddle test and, natural exposure test. A modified wicking test was developed for this
  project.
- 2. To develop a methodology to quantify efflorescence formation. Currently, the standard method is based on a basic visual observation criterion. For this study a quantification system will be established that includes image acquisition, and image analysis with the help of MATLAB to accurately reflect the severity. A MATLAB source code will be created to numerically output effloresced area percentages from an input image.
- 3. To evaluate the severity of efflorescence for early age carbonated samples and reference hydrated samples. The image analysis quantification system is used to compare the effloresced area of the carbonated and hydrated pavers.
- 4. To understand the carbonation effect on paver absorption through comparative evaluation, and its correlation to improved efflorescence resistance. Both semi and fully immersion absorption tests are performed on hydrated and carbonated paver samples.

- To determine the chemical composition of the efflorescence deposits through X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) coupled with Energydispersive X-ray spectroscopy.
- 6. To evaluate early age behavior of carbonated pavers, including strength gain and carbon dioxide uptake.

# **Chapter 2 Literature Review**

#### **2.1 Concrete Efflorescence**

Efflorescence usually develops on the surface of porous construction materials, normally as white, fine, lose and powdery, with color impression and appearance varying along with the level of severity (Brocken and Nijland 2004, Cement Concrete & Aggregates Australia 2006, Weng, Lin et al. 2013, Allahverdi, Najafi Kani et al. 2015). According to ACI 116R, cement and concrete terminology, efflorescence is defined as a deposit of salts, usually white, formed on a surface, the substance having emerged in solution from within either concrete or masonry and subsequently been precipitated by evaporation (ACI Committee 116 2000). Efflorescence normally can be found on both vertical and horizontal surfaces exposed to outdoor environment (Adam 2002). Its resulting color contrast is more pronounced on darker colored concrete elements from an optical point of view; conversely, efflorescence is hardly noticeable on white concrete (Kresse 1987, Moukwa and Vickers 1996, Aberle, Keller et al. 2001, Brocken and Nijland 2004). Iron oxide from the pigment will give efflorescence a yellowish tint; dirt and dust also have the ability to influence the appearance (Kompatscher, Ingrisch et al. 2007).

Unlike concrete discoloration due to inconsistency in materials and workmanship, efflorescence is not an indication of a structural defect, but regarded more of an aesthetic problem (Adam 2002, Dow and Glasser 2003, Brocken and Nijland 2004, Cement Concrete & Aggregates Australia 2006, Allahverdi, Najafi Kani et al. 2015). Nonetheless, efflorescence is an undesirable outcome, especially for landscaping projects, that can lead to the rejection of the product by customers, incurring significant financial and reputation losses (Cement Concrete & Aggregates Australia 2006, Delair, Guyonnet et al. 2007).

#### **2.2 Efflorescence Mechanism**

#### 2.2.1 General Overview

Efflorescence is a crystalline deposit of salts, containing either carbonates, sulfates or chlorides formed by the same mechanism (Kompatscher, Ingrisch et al. 2007). Dissolved soluble components from the material itself, or surrounding environment, migrate with pore water up to the evaporative surface (Aberle, Keller et al. 2001, Kompatscher, Ingrisch et al. 2007). The dissolved ingredients then either react to form insoluble salt (carbonate based efflorescence) or precipitate to deposits with the evaporation of the transporting media (Moukwa and Vickers 1996, Aberle, Keller et al. 2001, Behie and Chin 2010). Moreover, efflorescence can be influenced by the quantity and quality of the water within the concrete or exposed to it (Behie and Chin 2010).

The migrating solution moves through the concrete due to capillary suction, hydrostatic pressure, concentration gradients or gravity (Kompatscher, Ingrisch et al. 2007). Usually for efflorescence, soluble salts move to the surface as a result of moisture and/ or salt concentration gradients (Moukwa and Vickers 1996). Isberner (Isberner 1989) considered the evaporation force the most critical, a factor affected by material density. The denser a material, the harder it is for salts to move to the surface. three main factors control the evaporation force, and these are temperature, relative humidity, and air movement speed on the exposed surface (Kresse 1987).

#### 2.2.2 Primary and Secondary Efflorescence

There are two types of efflorescence based on the time of appearance; primary efflorescence and secondary efflorescence. Primary efflorescence normally tends to develop at the early age, usually days or weeks after service, with the carrying medium being water already in

the concrete. As for secondary efflorescence, white deposits start become visible months or even years after product application involving surrounding water from rain, dew, and/or drainage (Aberle, Keller et al. 2001, Adam 2002, Allahverdi, Najafi Kani et al. 2015). However, both of primary and secondary efflorescence follow the same mechanism of formation, mentioned earlier (Allahverdi, Najafi Kani et al. 2015).

#### **2.2.3 Chemical Composition**

The referenced study (Chin and Petry 1993) listed the composition of efflorescence deposits for 43 brick masonry wall samples collected from 24 individual projects in the US. Results revealed that 50% consisted of soluble sulfate, and 43% as calcium carbonate. Another study similarly revealed efflorescence deposits as mainly comprising sulfate salts or calcium carbonates after investigating 20 cases of clay bricks, as well as masonry and concrete blocks in the Netherlands (Brocken and Nijland 2004). Both the masonry material and the environment around can be the source for the soluble compounds that contribute to efflorescence formation (Sanders and Brosnan 2010). Table 2-1 from Chin and Petry's study shown below presents possible compositions of efflorescence for brick masonry wall, in order of frequency of occurrence.

Table 2-1: Possible chemical composition of efflorescence in order of most to least probable

Mineral	Chemical Composition
Calcium Carbonate	CaCO <sub>3</sub>
Sodium Sulfate	$Na_2SO_4$
Potassium Sodium Sulfate	$K_3Na(SO_4)_2$
Calcium Sulfate Dihydrate	$CaSO_4 \cdot 2H_2O$
Magnesium Sulfate Hydrates	MgSO <sub>4</sub> ·6H <sub>2</sub> O, MgSO <sub>4</sub> ·7H <sub>2</sub> O and MgSO <sub>4</sub> ·4H <sub>2</sub> O
Potassium Chloride	KCl
Sodium Chloride	NaCl
Sodium Hydrogen Carbonate Hydrate	$Na_3H(CO_3)_2 \cdot 2H_2O$

(Chin and Petry 1993)

#### 2.2.3.1 Carbonate Based Efflorescence

Calcium carbonate was initially believed to be the only form of efflorescence, which arises from the leaching of the cement hydration product  $Ca(OH)_2$  (CH). And its eventual reaction with atmospheric carbon dioxide. The dissolved CH is carried along with the pore water to the service surface, where it subsequently reacts with  $H_2CO_3$  (dissolved  $CO_2$ ) to form the insoluble salt  $CaCO_3$  (Aberle, Keller et al. 2001, Norsuzailina, Sinin et al. 2014, Allahverdi, Najafi Kani et al. 2015). CH concentration gradient in the transporting medium will drive more interior CH to move toward the reaction surface (Moukwa and Vickers 1996). Even the slightest amount of CH will trigger the reaction to form visible white deposits (Kresse 1987, Allahverdi, Najafi Kani et al. 2015). The overall chemical equation is shown below (Eq. 2-1) (Kresse 1989). The reaction can only take place in the presence of liquid water, given by :(Kompatscher, Ingrisch et al. 2007)

#### $Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$

#### 2.2.3.2 Sulfate and other soluble salt based efflorescence

In addition to calcium carbonate, sulfate salts are also detected in efflorescence deposits, however, chloride and nitrate salts only exist in some cases (Kresse 1987).

Control setting admixtures like gypsum, anhydrite and hemihydrate added during the manufacturing of cement usually serve as the source of sulfates (Chin and Petry 1993, Brocken and Nijland 2004). For chloride efflorescence, the components can originate from "1. Muriatic acid (hydrochloric acid) used to clean the brickwork. 2. Accelerant ("anti-freeze" compounds) in mortar. 3. Sea water either in the mortar (usually from sand washed in sea water) or deposits on the building in an ocean-side environment" (Chin and Petry 1993). Similar to the mechanism of carbonate based efflorescence, the soluble salts dissolve into the porous water, moving to the service surface as a result of the several possible driving forces mentioned earlier. These salt deposits don't further react like in the case of calcite formation, but instead directly precipitate with the evaporation of transporting media (Aberle, Keller et al. 2001). Salts deposits like sodium and potassium sulfate are very soluble, normally can be washed off by natural weathering after formation (Haage 1991).

#### 2.3 Laboratory Accelerated Efflorescence Test Methods for Concrete

All the accelerated efflorescence tests available involve exterior water as the transporting medium, therefore, based on the definition, these tests attempt to evaluate a product's potential for secondary efflorescence. There is no standardized efflorescence test for concrete, in fact, most researchers dealing with accelerated efflorescence for concrete adopt test method ASTM C67-14,

which is intended for brick and structural clay tiles. In recent years, researchers have been trying to create new test methods to accelerate the deposits-forming process, which usually takes months to occur for secondary efflorescence. Several feasible methods for both bricks and concrete are presented below.

#### 2.3.1 ASTM C67 for Bricks

McBurney and Parsons created an accelerated efflorescence potential test method called wick test in 1937 and used it on 684 bricks from 255 manufactures (McBurney and Parsons 1937). This method was then included into the National Bureau of Standards serving as the basis of ASTM C67. In this test method, samples are immersed in distilled water with a depth of 1 inch for 7 days in a room with a temperature of  $24 \pm 8$  °C and a relative humidity around 30 to 70%. These are then oven dried for 24 h to evaporate all the water inside. Appraisal is based on visual observation, where the observer inspects the tested sample against a reference (untested) one from a distance of 10 meter under a light illumination greater than 538.2 lm/m<sup>2</sup>. If a color difference is noticeable, then the sample is marked as "effloresced". Otherwise, the sample is simply denoted as "no effloresced" (ASTM C67-14 2014).

#### 2.3.2 Russian Efflorescence Test on brick

Behie and Chin compared the efficiency of efflorescence test from Russian and British to the ASTM C67 (Behie and Chin 2010). Similar to ASTM C67, the Russian efflorescence test also adopts a form of wicking test where water migrates from one side to the other (Fig. 2-1). Unlike ASTM C67, the Russian test modified the orientation and the service surface is exposed with the opposite side immersed. And it was found that both ASTM C67 and the Russian test had the ability to accelerate the efflorescence forming process with approximately the same efficiency.



Figure 2-1: Russian efflorescence test (Behie and Chin 2010)

#### 2.3.3 British Efflorescence Test on brick

For the British efflorescence test, instead of having water migrate upwards within the sample, it moves downwards from the top surface due to gravity. A two ends opened flask with 300 ml of distilled water sits on top the tested brick. A schematic is provided in Figure 2-2. This tests are conducted in a warm, well-ventilated room, and the amount of water maintained in the flask for 48 hours. The rest of the sides and bottom surface needs to be sealed. The water is then removed and the samples is dried for 7 to 9 days. The entire process is then repeated but the drying process extended for a period of 14 to 16 days (Institution 1985). In Behie and Chin's research, they found the British test was less efficient compared to ASTM and Russian Test. (Behie and Chin 2010)



Figure 2-2:British efflorescence test (Behie and Chin 2010)

#### **2.3.4 Wicking Test on Concrete**

The referenced work (Moukwa and Vickers 1996) used a wicking test to assess the efflorescence potential of cylindrical concrete samples. Impermeable epoxy was used to seal the side of the cylinders, but leaving 1 inch from the bottom exposed. Samples are then placed in suitable containers and filled with tap water to a level slightly below the top surface of the cylinders (Fig. 2-3). Water is continually added to maintain this level as a constant hydrostatic pressure increases the rate of diffusion of water through the concrete. Exposed surfaces are examined for efflorescence after 28 days. Moukwa and Vickers reported extensive efflorescence formation when observing wicked samples after 28 days of exposure (Moukwa and Vickers 1996)



Figure 2-3: Wicking test set-up (Moukwa and Vickers 1996)

#### **2.3.5 Percolating Test**

For this test, the entire side surfaces are epoxy sealed with a dike installed along the circular perimeter on the top surface shown in Fig. 2-4. Samples are kept upright in a vertical position and 10 ml of distilled water is poured into the dike. This is repeated once the bottom of the sample is no longer damp (Moukwa and Vickers 1996). Efflorescence was successfully produced by this method as reported by Moukwa and Vickers (Moukwa and Vickers 1996) but the formation speed was slower than in the wicking test.



Figure 2-4: Percolating test set-up (Moukwa and Vickers 1996)

#### 2.3.6 Wet-Dry Test Method on concrete

In this test method, samples are constantly subjected to wetting and drying cycles by being placed in a fog room for 16 hours, followed by 8 hours of drying. However, no noticeable efflorescence was formed after carrying this test our for one month (Moukwa and Vickers 1996).

#### 2.3.7 Puddle Test

The Puddle test was first presented by Kresse. It is an accelerated efflorescence test suitable for fresh and hardened concrete. Similar in principle to the percolation test, a puddle of water is formed on top of a sample, where the water then gradually penetrates through the sample. the dissolved efflorescence-related components will then migrate back to the surface with the subsequent evaporation of the absorbed water (Kresse 1987). This method has one drawback in that it necessitates the constant spraying of water on the surface, which in excess can wash away the dissolved salts or CH before forming any deposits. Kresse solved this problem by using dew to create a film of moisture on the surface. However, in order to have water condense on the surface, all the samples needed to be placed in a setting of low temperature and high moisture. In such an environment, the rate of evaporation, which is the most critical driving force for the migration of

efflorescence related components, will become slower. This consequently decreases the rate of salt deposition (Moukwa and Vickers 1996).

#### **2.4 Examination Methods**

The existing efflorescence test standard ASTM C67 includes an examination and rating method. This is based on a rudimentary visual observation, where an observer examines the top and all four sides of each sample and compare the superficial appearance with an untested reference sample from a distance of 10 ft. Examination needs to be carried out under an illumination of not less than 50 foot-candles (538.2 lm/m<sup>2</sup>). If any efflorescence can be observed, the sample is then identified as "effloresced". Otherwise, the sample is "not effloresced"(ASTM C67-14 2014).

Clearly, the ASTM test has no accurate quantification of efflorescence in its examination method. Only two ratings exist based on visually observation can lead to unavoidable intervention of human subjectivity. Therefore, several studies sought to establish a more precise method to quantify the degree of efflorescence. For example, Weng et al. used a MATLAB image analysis technique where an efflorescend area was differentiated with RGB values, and complemented by a curettage method including the collection of salt deposits with a spatula to determine efflorescence thickness (Weng, Lin et al. 2013).

Another image analysis treatment was established by Ferreira and Bergmann using Image Tool 3.0 (Ferreira and Bergmann 2011). A steel camera holder was designed as shown in Fig. 2-5 to keep a fixed focus distance of 170 mm from the sample. The picture was taken in an illuminated environment and the image was subsequently processed by the Image Tool 3.0 software. The

software first measured the area of the sample after removing the redundant background. Then the effloresced area as a ratio of the total area was determined.



Figure 2-5: Image Analysis Setup (Ferreira and Bergmann 2011)

Instead of turning the picture to black and white, Hennetier et al. developed a grey scale image analysis (Hennetier, Almeida et al. 2001). Pictures were converted into grey scale mode and a range from  $L_i$  to  $L_j$  was manually chosen to be the efflorescence contaminated range, where  $L_j$  is usually designated a value of 255. The degree of efflorescence is then approximated as the amount of pixels in the specified range with respect to the total pixels of the image.

#### **2.5 Efflorescence Removal Methods**

Nowadays, efflorescence deposits can be removed either by chemically or mechanically (Allahverdi, Najafi Kani et al. 2015). For chemical removal, the use of dilute acid like hydrochloric acid was proved quite effective, especially for carbonate based efflorescence (Kompatscher, Ingrisch et al. 2007). However, chloride acid agents were reported elsewhere to prompt chloride based efflorescence (Chin and Petry 1993). Furthermore, the surface color and desired texture may be damaged due to the acid (Allahverdi, Najafi Kani et al. 2015). This also represents only a temporary solution, as efflorescence can reemerge months after treatment (Aberle, Keller et al.

2001). As for mechanical removal, dry brushing is the most productive mechanical way to get rid of efflorescence (Kompatscher, Ingrisch et al. 2007). Sand blasting can also do the job and is usually applied for in service prefabricated concrete, like paving stones or blocks. Again, the texture and color may be altered (Kresse 1989). Other techniques include wet brushing or high pressure water jetting , if dry techniques proved ineffective (Kompatscher, Ingrisch et al. 2007). If water-bearing removal methods cannot solve the problem at once, efflorescence reappearance is quite probable with the evaporation of the water (Aberle, Keller et al. 2001). Both chemical and mechanical treatment will contribute to the aging of the structure.

If no treatment was applied to the contaminated surfaces, it is suggested that natural weathering will aid in the removal of light soluble deposits like sulfate based efflorescence (Kresse 1989, Hennetier, Almeida et al. 2001). This however is a lengthy process and it does not address insoluble efflorescence products like calcium carbonates (Chin and Petry 1993).

#### **2.6 Efflorescence Prevention Methods**

Efflorescence prevention methods should be designed based on the understanding of mechanism and sources of components. From the previous literature review, there are three main factors that play an important role in efflorescence establishment, and these are dissolvable chemical components, water as a transport media, and a pathway allowing the solution migrate to the exposure surface. The elimination of any one of these three factors can prevent the occurrence of efflorescence (Kompatscher, Ingrisch et al. 2007).

However, water and water soluble compounds are impossible to be completely avoided, several suggestions to minimize them are summarized from design and construction perspectives (ASTM C1400-11 2011). Rain water is believed to be source of transport media related to

efflorescence (Chin and Petry 1993). Therefore, design considerations should include fast drainage and ensure minimum water contact and penetration (Adam 2002, ASTM C1400-11 2011). Controlling moisture during service may also be beneficial in decreasing the number of wet-dry cycles as much as possible (Kompatscher, Ingrisch et al. 2007). With regards to the Raw concrete components, the use of low alkali cements is preferred, and all other contents including mixing water should be screened to have the least amount of soluble salts in them (Kompatscher, Ingrisch et al. 2007).

The intensity of efflorescence can also be reduced by minimizing the permeability and porous volume through either effective compaction, good detailing, sufficient cement use, optimized water and cement ratio, and pore-structure modifications to limit water movement (Adam 2002, Chang and Chen 2005, Allahverdi, Najafi Kani et al. 2015). High water permeable concrete could maintain the efflorescence potential for years (Kresse 1989). Pozzolanic material can be used as a supplementary cement additive to reduce efflorescence potential based on the following reasoning: first, the pozzolans consume CH, the primary source of carbonate-based efflorescence, through a pozzolanic reaction shown as Equation 2-2 and 2-3. Secondly, permeability and porosity are lowered because more pores become filled with the resulting extra C-S-H gel formed from the reaction (Norsuzailina, Sinin et al. 2014). Norsuzailina et al. reported that using the fly ash class F and silica fume as pozzolanic additives can effectively reduce efflorescence (Norsuzailina, Sinin et al. 2014). Other pore fillers that can reduce permeability include fumed silica, metakaolin, and limestone fillers (Aberle, Keller et al. 2001). Special additives also exist, e.g. Elotex ERA100 and Eflotex ERASEAL 120, whose primary role is to eliminate efflorescence through the "optimization of pore size, reduction of capillary connectivity and temporary reduction of  $Ca^{2+}$ , (Aberle, Keller et al. 2001).

Equations 2-2 and 2-3 show hydration of Pozzolans Modified Cement System, (Norsuzailina, Sinin et al. 2014).

$$C_3S + H \rightarrow C - S - H + CH$$
 Eq. 2-2

$$Pozzolan + CH \rightarrow C - S - H$$
 Eq. 2-3

Blend cement approach was presented by Constantinou in 2001, in order to avoid CH, additional calcium aluminate, calcium silicate, calcium sulphate and reactive silica added into the mix design (Constantinou 2001). The hydration equation for mix binder is given as Equation 2-4 (Constantinou 2001).

$$C_3S + CA + C\overline{S} + H \rightarrow C_3A \cdot 3C\overline{S} \cdot H_{32}$$
 (ettringite) +  $C - S - H$  Eq. 2-4

An exterior impermeable coating can sometimes be applied in order to protect masonry units from efflorescence. This is a costly prevention method for it requires additional material and labor (Aberle, Keller et al. 2001, Allahverdi, Najafi Kani et al. 2015). Moreover, efflorescence may precipitate beneath the coating, which is called cryptoflorescence or subefflorescence, and the cumulating deposits can cause disintegration of the surface (ASTM C1400-11 2011).

When curing the concrete in a constant and appropriate temperature and relative humidity, one should avoid drying, wind, drips and condensation (Kompatscher, Ingrisch et al. 2007). Concrete pavers have been found to effloresce during storage. When placed in pallets the concealed pavers were observed to experience extensive efflorescence, since water could not have been uniformly dried out. Therefore, uneven moisture condition during storage needs to be avoided (Kresse 1987).

#### **2.7 Carbonation Curing**

Anthropogenic carbon dioxide represents 83% of greenhouse emissions, contributing significantly to global warming. In 2013, CO<sub>2</sub> emissions in US was equivalent to 6,673 million metric tons (U.S. Environmental Protection Agency 2016). And globally 5% of CO<sub>2</sub> emissions originates from the cement industry, which is implementing more proactive efforts by encouraging the use of slag and fly ash for fractional substitution of cement in order to decrease the greenhouse gas emissions (Worrell, Price et al. 2001). Early-age carbonation curing of concrete also contributes to emission mitigation as CO<sub>2</sub> becomes permanently stocked inside the concrete. It has also been discovered to lend concrete several other technical advantages.

Applied concrete needs to survive different types of exposure attacks during service life, including, but not limited to, freezing and thawing cycles, chloride attack, sulfate attack, and weathering carbonation. Reactions related to weathering carbonation are summarized by Equation 2-5 and 2-6. Atmospheric carbon dioxide penetrate into the mature concrete and reacts with two main hydration products, calcium hydroxide and C-S-H (Groves, Brough et al. 1991). This associates a drop in pH as the CH is consumed, making reinforced concrete more susceptible to corrosion of the steel rebars (Parrott 1987). However, researches were reported that the weathering carbonation can densify the binding matrix and lower the porosity, and consequently decreasing the permeability of the concrete. The penetration of water contains harmful substances causing chemical attack can be reduced due to low permeability of concrete. (Zhang and Zong 2014) More

importantly, it also increases concrete's compressive strength and elastic modulus. (Richardson, Groves et al. 1993, Jerga 2004, Chang and Chen 2005)

$$CH + CO_2 \rightarrow CaCO_3 + H_2O$$
 Eq. 2-5

$$CSH + 2CO_2 \rightarrow SiO_2 + 2CaCO_3 + H_2O$$
 Eq. 2-6

With the observation that the weathering carbonation can increase the strength of the concrete and consume carbon dioxide. Early age carbonation curing of partially dried fresh concrete has been reported to considerably enhance concrete properties and result in rapid strength gain (Young, Berger et al. 1974, Shao and Lin 2011). During this process, the  $CO_2$  mainly reacts with  $C_2S$  and  $C_3S$  components of cement as presented in Equation 2-7 and 2-8 (Young, Berger et al. 1974). The gas initially requires an aqueous water medium to solvate in, which is served here by the pore fluid of the concrete. In order to fully optimize the diffusion of  $CO_2$ , a presetting of the concrete is necessitated since water content of freshly cast concrete contains is too high, thereby obstructing the gas' penetration efficiency. Therefore, for effective carbonation, fresh concrete is usually dried under controlled conditions until an optimum water content is achieved (Shao and Lin 2011). The main practical challenge for Carbonation curing is the cost of pure  $CO_2$  related to the carbon capture from emissions and its logistics.

$$C_3S + (3-x)CO_2 + yH_2O \rightarrow C_xSH_y + (3-x)CaCO_3$$
 Eq. 2-7

$$C_2S + (2-x)CO_2 + yH_2O \rightarrow C_xSH_y + (2-x)CaCO_3$$
 Eq. 2-8

# **Chapter 3 Experimental Procedures**

#### 3.1 Materials and mix proportion.

CAN/CSA-A3001 (Canadian Standards Association) Type GU ordinary Portland cement (Lafarge OPC) was used for this work. Granite was the aggregate material used for this project. To mimic Ducon's paver mix design, which consisted of top and bottom mixes, the top fine aggregates' gradation was strictly following Ducon's size distribution criteria presented in Table 3-1 and a corresponding sieve analysis graph is shown in Fig. 3-1. Scholz Black HS 50 was the pigment supplied by Ducon Industries for this project. The black colored pigment was chosen to increase the contrast of efflorescence. The mix design for a single paver is presented in Table 3-2.

Top and Bottom mix were prepared in two separate KitchenAid blenders simultaneously. Paving blocks with a dimension of 8cmx8cmx6cm were cast using the special made steel mold. The punch block was used for compaction of the pavers through an electrical vibration hammer (Fig.3-2). The bottom mix was put into the cavity of the mold first, and the concrete was hand compressed three times while filling the mold. Then using the vibration hammer to compact the concrete with a punch block for 15s. After that, all the top mix was filled in and compacted by hand before using a hammer to compress another 15s. Hand compaction can increase the quality of densification of the pavers and the levelness of the top surface.



Figure 3-1: Ducon's Top Fine Aggregates Sieve Analysis

T 11 A 1 A'		
Table 3-1. Steve	Analysis nerce	ntage naccing
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Sieve Size [mm]	5	2.5	1.25	0.63	0.315	0.16	0.08
% Passing	100	99.90	90.47	58.41	36.43	22.75	14.28

Table 3-2: Mix Design for a single paver

Materials	Aggregate						
	Тор	0-	5-	Cement(OPC)[g]	Water[g]	Pigment[g]	Total[g]
	Fine	5mm[g]	10mm[g]				
Top Mix	153.8	-	-	42.7	13.7	0.8	211.0
Bottom Mix	-	400.8	172.6	103.4	34.3	0.0	711.1


Figure 3-2: Manual compression and final paver sample

## **3.2 Carbonation Curing**

## **3.2.1 Precondition Drying**

Preconditioning drying was carried out with the aid of a fan and prior to carbonation curing in order to make more pore space for better CO<sub>2</sub> diffusion, and, therefore, reaction. After casting, the pavers were moved to a room with constant temperature and humidity (24 °C, 50%RH) for this presetting step. A pedestal fan was placed in front of the samples, and adjusted to provide uniform air flow to all pavers. Therefore, it is fair to assume that all samples were subject to the same evaporation rate. Drying was halted once the blocks achieved a 35 % loss of initial water content (i.e. mix water). A paver from each batch was placed on a balance and monitored for weight loss in order to achieve the target weight, after which the blocks are submitted to carbonation curing.

### 3.2.2 Flue Gas and Pure CO<sub>2</sub> Carbonation

The carbonation set-up is shown in Fig 3-3. After precondition drying, the specimens were weighed to gain reference mass and then placed into the chamber. Carbonation was carried out by pressurizing the chamber with  $CO_2$  up to 5 bar and maintained for 4 hours. A regulator ensured that the pressure remain constant throughout. Upon completion, a paver is retrieved and its weight were measured again. The paver specimens were then stored in a mist room (100% RH) up to 28 days for further hydraulic reaction of cement.

Two different purities of  $CO_2$  gas were used for carbonation, 99.5% and 20%, for pure  $CO_2$  carbonation and flue-gas CO2 carbonation, respectively. Pure gas is more effective but could be costly. Flue gas is a much cheaper option. It can be easily collected and used directly without significant capital cost. However, the efficiency of flue gas carbonation reaction could be reduced.



Figure 3-3: Carbonation curing set-up

#### 3.2.3 Quantification of CO<sub>2</sub> Uptake

According to the mass gain method presented by Monkmand et al., CO<sub>2</sub> uptake is a ratio of the mass of CO<sub>2</sub> sequestrated by the specimen as a fraction of the initial mass of cement added in the mix design (Monkman and Shao 2006). Before and after carbonation weights were measured, and the weight increase is primarily due to the fact that carbon dioxide dioxide's engagement with CH and calcium-silicates to form calcium carbonates. Water vapor emanating from the reaction was was collected using paper towels from the bottom, lip, and sides of the chamber's interior after carbonation, as this weight is to be accounted for as part of the specimen's final mass. All specimens were assumed to have lost the same amount of water during carbonation. The CO<sub>2</sub> uptake for each specimen was calculated by dividing the difference between the final and initial mass by the total amount of cement used in the mix design for each paving block. The formula used:

Carbon Uptake (%) = 
$$\frac{(W_{After \ carbonation} + W_{Water \ lost}) - W_{Before \ carbonation}}{W_{Cement}} \times 100\%$$
 Eq. 3-1

Where  $W_{After\ carbonation}$  is the weight of the specimen after carbonation,  $W_{Water\ lost}$  is the water retrieved from the chamber,  $W_{Before\ carbonation}$  is the weight of the specimen before carbonation, and  $W_{Cement}$  is the weight of the dry cement used for a specimen.

## **3.3 Compression Test**

Compressive test conducted on four samples were all at an age of 24 hours. For two carbonated pavers, after 4 hour pure CO<sub>2</sub> carbonation curing, another 20 hours of subsequent hydration in mist room was performed. And for hydration samples, they were kept in mist room for 24 hours after casting.

Compressive strengths for the pavers with different curing method were measured by a MTS rock compression machine. First, the dimensions of load surface of each sample were assessed with a digital Vernier caliper. After that, each paver was placed under the pressure steel plate and loaded at a rate of 0.5mm/min until failure. The maximum load was recorded and the compressive strength can be calculated using the equation shown below, given by: (Novak and Colville 1989)

$$R_b = \frac{F}{A_c} \left[ N \cdot mm^{-2} \right]$$
 Eq. 3-2

where:

R<sub>b</sub>--- Compressive Strength

F--- Destruction Force in N

## **3.4 Evaluation of Efflorescence Potential**

### **3.4.1 Sample Preparation**

Three batches were cast including pure CO<sub>2</sub> carbonated paver CWC, flue gas carbonated FCWC and hydration cured batch HWC. For carbonated pavers, after the carbonation curing process described in section 3.2, all the cured paving specimens were moved into the mist room (25°C, 100% RH) for 28 days subsequent hydration.

Hydration curing was conducted by placing paving blocks into a 100% Relative Humidity hydration chamber right after casting for 28 d. This type of curing technique is the conventional method, and the hydrated pavers will be used as the references throughout the project.

Sample preparation was carried out in accordance with ASTM C67. After 28-day subsequent hydration in the 100% RH mist room, all the pavers (carbonated and hydrated samples) were weighted and oven-dried at 110 to 115°C for not less than 24 hours drying. Specimens were regarded completely dry when the difference between two successive measurements taken at 2-h intervals presented a difference not greater than 0.2% of the last scaled weight. The dried pavers were then placed in a temperature and humidity controlled room (24°C; 50% RH) for cooling the samples back to room temperature for not less than 4 h before any test. All the specimens were stored and tested in this certain room.

## 3.4.2 Efflorescence Quantification Method

#### 3.4.2.1 Image Acquisition

Image capture is the most critical step for image analysis, so it is important to ensure the process is consistent and repeatable (Hennetier, Almeida et al. 2001). Two pictures were taken before and after the efflorescence test for each specimen. Moisture can vary the appearance and affect the image analysis results, therefore, a process of 24 h furnace drying and 4 h cooling was always performed before taking images to ensure same moist level. All the images were captured in a dark room with unvarying light supply. Locations for specimen and camera were adjusted and marked. All the samples were photographed at the same location with a fixed distance of 120mm from the camera. One set of accurate exposure settings of the camera were used for all pictures. Exposure time: 1/30; ISO 125; Focal length: 4.15; FNumber: 2.2. With the intension of capturing all the edges of the paver in the picture, a stopper was used to lift the specimen. Image acquisition is demonstrated in the schematic of Figure 3-4.



Figure 3-4: Image acquisition set-up

#### 3.4.2.2 MATLAB Image Analysis

The background for each picture was reset to white using Photoshop, and the detailed perimeter of paver was kept. Modified pictures were imported into MATLAB and turned into black and white images. A luminance level was used as a threshold in this process, which means each pixel in the image will be turned into either 1 for white or 0 for black depending on whether it passes the threshold or not. After several trials, 0.5 was determined as the luminance level which can accurately identify the efflorescence area and turn other parts to black.

The threshold was set to 0.99 first to isolate the white background. The amount of black pixels, denoting the surface area (A), and the amount of white pixels, relating white background (BG), was determined by MATLAB. The color picture was then turned to black and white again, but with a luminance level of 0.5, and the effloresced area can now be determined in pixels by subtracting the white background (BG) from the amount of total white pixels. A MATLAB script was designed to accomplish the mentioned tasks and output original and black-white images.

#### **3.4.3 Evaluation of Efflorescence Testing Methods**

No standard test method exists for assessing efflorescence potential for concrete pavers, and some researchers adopted ASTM C67, the standard efflorescence test for brick and clay tile, when they were working with concrete and mortars. A preliminary test was conducted using ASTM C67 on hydration concrete pavers. No efflorescence was observed after the test, suggesting that the standard test for brick and clay tile is not suited for concrete paver samples. Therefore, a new modified wicking test was designed based on C67 with severe exposure conditions. Two more test methods are also evaluated. They are puddle test and natural exposure test. For tests

assessments, hydrated specimens were only used for the purpose of accelerated efflorescence generation and pacing the rate of observable generation.

#### 3.4.3.1 Modified Semi-Immersed Wicking Test with Capillary absorption rate monitored

ASTM C67 is a standard efflorescence test method for brick and clay tiles, but it is not efficient for concrete. The modified wicking test was based on this standard with some applied modifications were applied to aggravate the test conditions (Fig. 3-5). Six pavers, two from each batch (CWC, FCWC, HWC), were weighted one by one and placed onto two rubber hollow circular stoppers to increase the bottom surface water contact area. Choosing the smallest possible size of containers helped minimize excess salt leaching from the paver. Each specimen was placed in a separate container. Isberner considered the evaporation forces as the most critical factor for efflorescence formation. (Isberner 1989) Therefore, besides temperature and humidity which are constant at 24°C and 50% RH in this case, air flow is also an influencing parameter. A fan was set in front of the pavers and adjusted to make each sample exposed to relatively the same air flow speed (Fig. 3-6). The wind speed was recorded at 7.6 km/hr. at the top surface by a Kestrel environmental meter. The containers were filled with distilled water until the pavers was immersed by 25mm. The water level was maintained by frequent replenishing throughout testing. Distilled water was added twice a day, at 9am and 5pm.

Additional 24 hours 110°C furnace drying was conducted after running the accelerated efflorescence test for 10 days. The environmental controlled room used again for cooling the dried pavers back to 24 °C before the pictures were taken.

The semi-immersed absorption rate was calculated by recording the surface-dried weights scaled according to the schedule of mass measurements, the pavers were removed away from the

containers and surface-dried using brown paper towels before weighing the mass. The measurements were taken at (time after contacting with water): 30 s; 1 min; 5 min; every 5 min up to 30min; 45 min; 60 min; 1.5 h; every hour up to 7 h; once a day up to 8 days.

Four more pavers were cast to double check the results. Two of them were carbonation cured and the other two are hydration reference. Only modified wicking test was performed and the efflorescence severity was analyzed.



Figure 3-5: Schematic of modified wicking test set-up



Figure 3-6: Modified wicking test set-up

## 3.4.3.2 Puddle Test

Puddle test, also called percolating test, is an efflorescence potential evaluation test that can be conducted on both fresh and hardened concrete. As the name implies, it involves creating a puddle of distilled water on top of the paver, and the water allowed to penetrate. (Kresse 1987)

A piece of rubber, used as a dike to seal a perimeter, was cut to fit the size of the top surface. Two steel holders were applied to grip the concrete paving block and provide adequate pressure for the rubber to hold the water (Fig.3-7). 20ml of water was added each time to the top to dampen the underside of the sample. Incremental additions of 20ml were needed once the bottom side of the paver is no longer damp.

As in the case of the modified wicking test, additional oven drying and cooling steps were similarly performed before taking images for MatLAB image analysis.



Figure 3-7: Puddle test set-up

### 3.4.3.3 Natural Exposure Test

In addition to the lab tests, eighteen differently cured pavers were cast an exposure test to experience real-life wet-dry cycles. The three batches of pavers were hydration-cured, pure CO2 cured, and flue-gas cured. Ducon's installation guide for standard paver-laying was followed (Ducon Industry FZCO 2012).

In consideration of mobility, the pavers were laid into a plastic box with dimension of 60cmx 60cm x 40cm. Several drainage holes were drilled on the side and bottom of the box. Laying entailed a number of material layers, and each were marked for depth on the side of the box. The respective layers of each material is presented in Fig. 3-8 and 3-9. A layer of geotextile was placed first to protect the soil from sweeping away by draining water. A 5cm layer of compacted soil was filled and covered by another layer of geotextile to prevent plant growth. The entire assembly was placed on a shaking table to help level the sand, and other material layer. Next, a 2-inch layer of 0  $-\frac{3}{4}$  inch gravel base was laid, racked smooth and compressed before putting another 2 inch were

similarity piled on top. This was repeated until a 12cm layer of gravel base was achieved. A layer of bedding sand was then piled and leveled on top of base gravel to form a thickness of, 3.5cm The pavers were then laid on the bedding sand with consistent joint widths. Finally, polymeric sand was spread on to the paver and swept into the joints. Water was sprayed on the pavers to densify and activate this joint sand. The final set-up presented in Fig. 3-10. An electrical vibratory hammer was used throughout to ensure proper compaction and levelling on individual material layers. Finally, a steel mesh was used to cover the entire box to protect against tampering by animals.

A location was chosen on campus allowing representative climatic exposure, where the pavers were not obstructed by surrounding buildings in order to have them sufficiently exposed to sunlight, rain, and wind. Fig. 3-8 illustrates each layer of setting materials.



Figure 3-8: Paver installation (Ducon Industry FZCO 2012)



Figure 3-9: Paver installation with depth of each layer



Figure 3-10: Natural exposure test

#### **3.4.4 Fully Immersed Absorption and Permeable Voids Test**

The test was conducted in accordance with ASTM C642, the standard test method for density, absorption, and voids in hardened concrete (ASTM C642-13 2013).

After initial curing and subsequent hydration of 28 days, concrete paver samples were dried in an oven at 110°C for 24 hours. Then, the samples were allowed to cool down in a 24°C room. After cooling, mass of each specimen was determined. Next, all the pavers were subjected to an additional 24 hours of oven drying. Room temperature weights were verified again to make sure the difference between the two successive values did not exceed 0.5% of the smaller value. The second measured masses were considered as the paver's oven-dry mass, annotated 'A'.

The dried pavers were fully immersed in distilled water with a temperature around 21°C for 48 hours. The surface dried masses were measured before and after placing them back into water for another 24 h period of immersion. Two consecutive values were validated to ensure that the increase in mass is less than 0.5% of the larger value. these larger values were assigned as a paver's immersion mass, 'B'.

After immersion, the soaked pavers were placed into a cooking pot and fully submerged in distilled water. The pot was brought to a boil by using a hot plate and continued for 5 hours. The pot was then removed and placed in ambient conditions to recede back to room temperature. The surface dried masses of the cooled pavers were determined as the boiled masses, 'C'.

Boiled pavers were placed in a steel basket and immersed in water. The immersed apparent mass for each paver 'D' was determined by weighing the suspended basket. The absorption and the volume of permeable voids can be calculated using Eqn.3-3 to 3-5 (ASTM C642-13 2013).

Absorption after immersion,  $\% = \left[\frac{B-A}{A}\right] \times 100$ ; Eq. 3-3

Absorption after immersion and boiling, 
$$\% = \left[\frac{C-A}{A}\right] \times 100$$
; Eq. 3-4

Volume of permeable voids, 
$$\% = \left[\frac{C-A}{C-D}\right] \times 100$$
; Eq. 3-5

Where:

A = mass of oven-dried sample in air, g

B = mass of surface-dry sample in air after immersion, g

C = mass of surface-dry sample in air after immersion and boiling, g

D = apparent mass of sample in water after immersion and boiling, g

## 3.4.5 X-Ray Diffraction (XRD)

XRD is an effective diffraction analytical technique, conducted to determine the chemical composition of efflorescence formed during the wicking test. A Bruker D8 Discover system with a 2D Vantec detector was used, capable of scanning a predetermined area. In principle, it is a focused X-ray beam that irradiates a sample's surface, interacting with the crystalline portion of a sample. Diffracted X-rays are collected by a detector and converted to information pertaining the crystal features of minerals present in the sample. The irradiation angle covers a specified range on order to get a complete spectrum with intensities and angle positions. The mineral composition can then be identified with the aid of the ICDD (International Centre for Diffraction Data) database.

A top corner of the hydrated paver and carbonated paver specimens were chiseled off

manually by hammer and chisel. The hydrated paver top corner was carefully chosen to display areas of both effloresced non-effloresced. A spot analysis of the two areas was then carried out via in-situ XRD analysis, where spot 1 was that of the effloresced area and spot 2 that of non-effloresced area. The Fig. 3-11 represent the prepared sample and also a close look of spot 1 and 2.



Figure 3-11: Top corner of hydrated paver specimen being subjected to spot XRD analysis

## 3.4.6 Scanning Electron Microscope with EDX Analysis System

Hitachi SU-3500 Variable pressure Scanning Electron Microscope was used for the surface analysis and high magnification image capture. Energy Dispersive X-ray analysis was also conducted to determine the elemental composition. The accelerating voltage used was 15 KeV and the images were captured at a magnification of x5.0k.

Efflorescence features generated in both the hydrated and carbonated specimens were closely examined here, morphologically and compositionally.

## **Chapter 4 Results and Discussion**

### 4.1 Evaluation of Efflorescence Testing Methods Using Hydrated Pavers

As mentioned in Chapter 3, three potential efflorescence tests for hydrated concrete pavers were conducted. The two accelerated tests, the modified wicking test (based on ASTM C67) and puddle test (based on research from Moukwa's research)(Moukwa and Vickers 1996), were conducted under laboratory conditions. The last test was the natural environmental exposure method devised to mimic real-life service conditions. This preliminary evaluation was carried out on hydrated-only paver specimens (cured in mist room, RH = 100%).

For the modified wicking test, dampness atop the samples was first observed after approximately 3 days of being semi-immersed in distilled water, indicating that the pores were fully saturated by this time, where the water evaporable front reached the uppermost extremity of the pavers. Extensive white efflorescence became visible atop the pavers and also at the sides after 6 days. Subsequent 24-hour drying (110°C) did not intensify efflorescence visibility after 10-day test. For the puddle test, no obvious white spots were detected after a 7-day testing cycle. After three more cycles (7 days/cycle), totaling 28 days of testing, only a tiny area of efflorescence was observed on the sides of the samples. Finally, for the natural exposure test, no efflorescence was observed on surface or at the sides of the pavers after undergoing six months of real-life wet-dry cycling. This test commenced in May and was ended in November.

All five sides of the pavers' surface (one top layer and four sides) were analyzed using a code-generated approach by means of MATLAB software, where images were converted to black and white based on a luminance level of 0.5. White regions were carefully adjusted to represent

efflorescence, and supported by visual observations. To quantify the intensity of efflorescence, the averaged white area percent for each batch was calculated and shown in Fig. 4-1. The modified wicking test seemed to prompt the highest degree of efflorescence on the pavers; 4.62% of the top area and 12.62% of the total area of the sides was determined effloresced. The output images are presented in Fig. 4-2 to 4-5. For the puddle test, no efflorescence was observed on the top surface and only 1% of the area of the sides was quantified as effloresced. Natural exposure did not result in observable efflorescence generation, even after a period of 6 months.

Based on the above evaluation, the modified wicking test was found most effective in accelerating the efflorescence formation in concrete pavers. Considerable amounts of efflorescence were observed after 10 days of testing. Areas with the most intense color disparity before and after testing were accurately identified as "effloresced" and turned into white in the devised black and white image analysis. On this basis, both the modified wicking test and the study's image analysis approach were adopted as the evaluation tool to test if carbonation curing can reduce efflorescence as hypothesized.



Figure 4-1: Efflorescence Area Percentage in hydrated concrete pavers (WH: wicking test and

PH: puddle test)



## Figure 4-2: WH Top Surface Image Analysis



Figure 4-3: WH Top Sides Image Analysis



Figure 4-4: PH Top Surface Image Analysis



Figure 4-5: PH Sides Image Analysis

## 4.2 Carbonation behavior of concrete pavers

The CO<sub>2</sub> uptake for each carbonated sample was calculated through mass gain method, a ratio of the mass of CO<sub>2</sub> sequestrated by the specimen with the mass of cement, using Equation 3-1, and the results are shown in the Table 4-1. After 4 hours of pure CO<sub>2</sub> curing at a pressure of 5 bar, the average CO<sub>2</sub> uptake reached up to 10.38% of the weight of cement. However, for flue gas  $(20\% \text{ of } CO_2)$  carbonation under the same pressure, the average uptake was only equivalent to 3.29 wt% cement.

Low uptake was expected for flue gas carbonation due to low  $CO_2$  concentration. Lower uptake indicates less calcium carbonate formation in the pores. Therefore, the pure  $CO_2$  carbonated pavers were predicted to have better resistance to efflorescence.

Name	Conditions	CO <sub>2</sub> Uptake	Average[%]	Standard Deviation
CWA-1		11.10		
CWA-2		10.70		
CWC-1	100% CO <sub>2</sub> ; 5bar; 4h	10.83	10.38	0.45
CWC-2		9.85		
RCWC-1		10.37		
RCWC-2		9.97		
C1-1		10.07		
C1-2		10.15		
FCWC-1	20% CO <sub>2</sub> ; 5bar; 4h	3.34	3 29	0.078
FCWC-2		3.23	5.27	0.070

Table 4-1: CO<sub>2</sub> Uptake for carbonation curing

CWA: Pure CO<sub>2</sub> carbonated pavers for fully absorption test

CWC: Pure CO<sub>2</sub> carbonated pavers for semi-absorption test and modified wicking test

FCWC: 20% CO<sub>2</sub> carbonated pavers for semi-absorption test and modified wicking test

RCWC: Repeated batch, pure CO<sub>2</sub> carbonated pavers for modified wicking test

C1: Pure CO<sub>2</sub> carbonated pavers for compression test

## **4.3 Compression Test**

The average results from the compression test conducted on four samples are shown in Fig. 4-6, with carbonated pavers referred to as C1 and hydrated reference referred to as H1. The compressive strength of the batch C1 corresponding to the curing regime of 17 hours' hydration curing followed by 3 hours of presetting and 4 hours of carbonation is 28.61MPa, which is higher than 24-hour hydration reference with a strength of 21.76Mpa. Therefore, pure CO<sub>2</sub> carbonation process was able to increase the compressive strength by 31.5% compared to hydrated paving specimens.

One of the advantages of carbonation curing confirmed was that it can provide faster strength gain. Therefore, this technique can reduce the production cycle and provide more flexibility to manufactures to arrange storage and transportation.



Figure 4-6: Compressive Strength

## 4.4 Accelerated Efflorescence Wicking Test and Image Analysis

Under the adopted experimental conditions, the modified wicking test proved most suitable for testing the potential of efflorescence formation for the paver specimens, while the MatLAB image analysis was able to accurately quantify the area percentage of the contaminated area. The test and analysis served as the primary indicators for carbonation curing's effect on efflorescence performance. Three different batches were tested: the pure carbon dioxide carbonation cured (CWC); flue gas carbonation cured (FCWC); and hydration cured reference samples (HWC). Except the curing method, the mix design, cast and prepare process were identical for all of the pavers.

Original paver surface images and their MatLAB outputted black & white rendering are shown in Figures 4-7 to 4-9. Clearly, no noticeable efflorescence can be observed on the top surface for carbonated pavers (batch CWC) after undergoing wicking test for 10 days, with the superficial appearance kept the same. On the other hand, extensive white and yellowish deposits were formed on the surface of the hydration reference (Fig. 4-9). As for the FCWC batch, with 20% CO<sub>2</sub> flue-gas carbonated pavers, efflorescence could not be eliminated completely (Fig. 4-8). The crystalline deposits of salts were found underneath the very top surface of FCWC, but the deposits were still noticeable and a large area was determined as "effloresced" by image analysis. For hydration reference HWA, significant efflorescence was observed on top surface. Parallel comparison of facial appearance after testing between CWC, FCWC and HWC is presented in Fig. 4-10. Fig. 4-11 summarizes the ratio of effloresced area with respect to the total surface area. The highest effloresced area was 11.42% and belonged to the hydration reference pavers, compared to 0.14% for the carbonated pavers. For the latter, the 0.14% increase in white pixels may be due to slight appearance alterations resulting from weathering and wet and dry cycling. Flue gas carbonation yielded an average 9.11% efflorescence affected area for the examined pavers.

An observation worth noting, and illustrated in Fig. 4-12, is that for the carbonated pavers (CWC batch), although no efflorescence was observed on top of the pavers, a line of white deposits formed below the colored layer at approximately 0.5cm above the water level in the wicking test. This line indicated the location for salt precipitation and referred to herein as the "deposits front", observable on all three batches. The front extended to the top black layer in the FCWC batch, and this is what influenced the higher overall effloresced area percent for these pavers (9.11 %), where the top surface alone accounted for 5.45% of the total inspected area. Finally, for hydration reference batch HWC, the deposits front was observed at the very top of the pavers.

Based on the mechanism of efflorescence formation, the soluble salts will precipitate with the evaporation of the transport solution. Therefore, the location of the deposits front is the same as the evaporation front. Seemingly, pure CO<sub>2</sub> carbonation curing can hinder the migration of the pore solution because of the densification from calcium carbonate precipitation. The degree of carbonation is expected to be higher for the top layer due to a higher cement fraction in the mix design (Table 3.3), and therefore a more pronounced densification effect arising from CaCO<sub>3</sub> precipitation. This subsequently results in a sort of capping effect that obstructs the upward wicking of solution. Therefore, instead of evaporating at the top, the water can only moved 0.5cm above the water level for the CWC pavers. The soluble salts could only deposit at this lower evaporation front on the four sides of these pavers. At this location, the primary efflorescence driving force, evaporation force, was much smaller than the top surface due to smaller exposure area and lower wind speed. And the evaporation force will be much smaller for sides of paver after

installation, because pavers will be immersed by joint sand and only the top surface is exposed to the open air. Conversely, the flue gas (20% CO<sub>2</sub>) carbonated blocks underwent a lesser degree of carbonation, as reflected by the low CO2 uptake recorded for these blocks of 3.29% compared to 10.38% for pure CO<sub>2</sub> cured samples. The evaporation front for the FCWC pavers seemed to migrate more freely upwards, where flue-gas carbonation was able to partially limit the complete wicking of the solution, but not sufficient enough for efflorescence to be nonvisible. It seems like the densification from 3.29% uptake was not adequate to prevent water from moving up to the visible surface and resulting in detectable sub-efflorescence. The evaporation front for the hydrated pavers (HWC) can be seen just below the service surface.

As previously stated, pure  $CO_2$  carbonation curing successfully prevented the samples from efflorescence formation compared to extensive efflorescence formed on the reference hydration pavers experienced by the modified wicking test. Flue gas carbonation does have some positive effect on efflorescence control, but the salts were still noticeable on the top surface. Furthermore, the efflorescence is merely considered as an asthmatic problem and it does not lead to mechanical damage (Adam 2002). So the efflorescence can be controlled at the invisible lower portion.



Figure 4-7: Pure CO2 Carbonation Curing Sample Image Analysis CWC



## FCWC1

# Figure 4-8: 20% CO2 Carbonation Curing Sample Image Analysis FCWC



Figure 4-9: Hydration Curing Sample Image Analysis HWC



cwc

FCWC

HWC

Figure 4-10: Parallel Comparison



Figure 4-11: Efflorescence Area Percentage



Figure 4-12: Deposits Fronts for each batch

The efflorescence formation tests were repeated to verify the test method and assess the effect of carbonation curing before the conclusions can be made. Two more batches were cast and labeled as RCWC for carbonated pavers and RHWC for hydration reference. It should be pointed out that new bag of pigment was used in these two batches. The new pigment supposed to be identical with the first one, since both of them were delivered by Ducon. But the new pigment was found to be less efficient compared to the previous one, and it might lead to difference in results.

After the pavers underwent the second 10-day modified wicking test, the same phenomena were observed as the previous test: there is no efflorescence on RCWC batch but noticeable white tint contamination on hydrated reference pavers as shown in Fig. 4-13. Image analysis determined that 2.93% of the area of hydrated pavers were covered by efflorescence, and only 0.11% for RCWC may be because of weathering (Fig. 4-14). Moreover, from Fig. 4-15, the deposit front was also observed on the sides at the lower part of RCWC. The intensity of the efflorescence was different when comparing the results from HWC and RHWC. The inconsistency on pigment and manual compaction may be responsible for the varying results. However, in general, hydrated pavers always showed clearly white tint on top and no trace of efflorescence on the carbonated pavers.





Figure 4-13: Image Analysis RCWC & RHWC



Figure 4-14: Efflorescence Area Percentage for RCWC and RHWC



Figure 4-15: Deposit Front for carbonated paver (RCWC)

### 4.5 Semi-Immersed Absorption Test

Water is one of the most important factors for efflorescence forming, since it is the transporting media to bring the components up to the surface (Kompatscher, Ingrisch et al. 2007). If the water pathway can be blocked by calcium carbonate precipitates formed within the pores during carbonation curing, then the problem of efflorescence can be eliminated. A lower evaporation front for carbonated pavers was observed compared to hydration reference, therefore, the absorption for CWC was predicted lower that HWC. An absorption test was conducted to assess such an effect by monitoring the amount of water absorbed by a block during wicking test. The surface dried weight measurements were taken at a schedule modified from ASTM C1585-11. Shorter time intervals between readings ensured a more accurate absorption rate profile. The absorption, reported in terms of percentage weight gain, was calculated using Equation 4-1.

Absorption 
$$[\%] = \frac{W_n[g] - W_o[g]}{W_o[g]}$$
 Eq. 4-1

where: Absorption [%] is the water absorption in percentage

 $W_n [g]$  is the surface dried weight after n seconds contacted to water

W<sub>o</sub> [g] is the original weight

Fig. 4-16 summarizes the average absorption data for each batch, and the slope of the curves indicates the rates of absorption. The most apparent observation is that the hydrated batch HWC and flue gas carbonated batch FCWC both display very similar absorption profiles, with the maximum absorption recorded as 4.0% after 14 hours of monitoring. Conversely, the pure  $CO_2$  carbonated batch had an average absorption of 2.7%. As hypothesized, pure carbon dioxide carbonated batch has much lower maximum absorption. It is in agreement with the observation

from wicking test that the water can fill the pores all the way up to the top surface for hydration reference, but no dampness was noticed on the surface of the carbonated CWC batch.

Each curve in Fig. 4-16 can be separated into two parts. The first part represents more or less a linear portion, which can be referred to as initial absorption. Beyond linearity, the curve gradually plateaus with relatively small changes in absorption, where this part of the curve is referred to as secondary absorption (ASTM C1585-13 2013). The rate of absorption for both HWC and FCWC decreased dramatically after 7 hours of semi-immersion, and almost became zero beyond that point. However, for the pure  $CO_2$  carbonated batch (CWC), the curve kept increasing beyond the 7 hours mark, recording a 0.6% increase in absorption, from 2.1% to 2.7%, in 17 hours. Therefore, the transition point between initial absorption to secondary absorption occurred somewhere between 7 hours and 17 hours for CWC.

Linear regression analysis was conducted using Excel with a trend-line was fitted to the linear portion of each curve. A linear regression equation was also estimated. The slope of this portion reflects the rate of absorption. Fig. 4-17 zooms in on the linear part of the curves. The HWC pavers displayed the highest initial rate of absorption at 0.0226 [%/sec<sup>0.5</sup>], while CWC displayed the lowest at 0.0122 [%/sec<sup>0.5</sup>]. Flue gas carbonated samples yielded a median rate of 0.0183 [%/sec<sup>0.5</sup>] between HWC and CWC.

To sum up, pure  $CO_2$  carbonation curing decreased the pavers' maximum absorption capacity to 2.7% compared to 4.0% for the hydrated reference pavers. Additionally, the CWC batch had the slowest rate of absorption as it took longer for it to finish the initial absorption stage. The 20% of  $CO_2$  carbonation curing failed to decrease the absorption rate according to the adopted testing procedures.



Figure 4-16: Semi-Immersed Absorption Rate


#### Figure 4-17: Initial Absorption Linear Regression

### 4.6 Fully Immersed Absorption and Permeable Voids Test

In addition to the semi-immersed test, a fully immersed absorption test was also performed to detect if the total voids volume was reduced by carbonation curing according to ASTM C642-06, the standard test method for density, absorption and voids in hardened concrete. Batch CWA, pure  $CO_2$  carbonated pavers, and reference hydration batch HWA were cast for this test.

The fully immersed absorption test aimed to find out if carbonation curing can decrease the total permeable voids volume and diminish the complete water absorption at the same time. Only 75% of the pores can be filled using cold water immersion; therefore, the boiling water method was applied to drive the water fully to the interior in order to achieve complete absorption by the pores (Chandigarh 2001).

The measured absorptions are displayed in Fig. 4-18. The hydration reference pavers consistently exhibit higher water absorption values by weight than those for the carbonated pavers. Submersion in boiling method increased the penetration of the water such that the complete water absorption for the hydrated pavers measured as 6%; and 5% for CWA, a decrease of 19% as a result of carbonation curing. Similarly, carbonation curing was also found to reduce the permeable voids volume by 1.9% according to Fig. 4-19. And again, the percentage decrease of voids volume was calculated as 13.7%.

Comparing to hydration reference, pure  $CO_2$  carbonation curing decreased both of the permeable voids volume and the fully immersion absorption. It is another indication that the

carbonation curing can slow down the water movement through concrete densification, and the resistance to efflorescence of carbonated pavers was increased consequently.



Figure 4-18: Absorption after Immersion and Boiling



Figure 4-19: Volume of Permeable Voids

## **4.6 X-Ray Diffraction (XRD)**

X-Ray Diffraction was used to identify the mineral form of efflorescence formed on the hydration cured pavers, and its chemical composition. Two spots were targeted during in-situ analysis of the hydrated paver: 1. area without efflorescence, referred to as "Hyd. TopNoEff," and 2. area with efflorescence, referred to as "Hyd. TopEff," respectively labeled as spot 1 and spot 2 respectively. Both spots located on the top surface of the paver. The XRD spectra collected are shown superimposed in Fig. 4-20 below. Pattern matching was carried out with the help of PANalytical X'Pert HighScore software with the highest probable match shown in the reference spectrum of Fig. 4-21.

During analysis, the x-ray beam's interaction zone is not confined to the analyzed spot alone but also interacts with neighboring components which become reflected by foreign peaks in the spectrum. The spectrum of spot 2 may very likely include crystallographic information pertaining to the concrete below the probed deposit layer. Peaks related to efflorescence can be deduced by simply subtracting the spectrum of spot 2 from spot 1. There are four peaks at 29.8°, 30.8°, 31.0° and 37.2° exist in spot 2, but not in spot 1. Through software pattern matching on these four peaks, potassium sulfate was identified as the main salt.

Since the test directly scanned a fractured part of the concrete paver, rather than a powdered sample (powder diffraction analysis), a considerably amount of background peaks with relatively low intensities exist. Peaks shifts, intensity count, and preferred crystal orientation, are all spectral features that may be more pronounced when conducting in-situ spot analysis. The detection of quartz (peak at 26.7°) was prevalent during analysis which originated from the sand component of the concrete mix design.

SEM was employed as a complementary analytical technique to lend more details related to the morphology and composition of the observed efflorescence.



Figure 4-20: XRD spectra for the hydrated paver fragment showing spots 1 and 2





Figure 4-21:Reference pattern for potassium sulfate

# 4.7 Scanning Electron Microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX)

The SEM system coupled with EDX is an extremely powerful technique for surface characterization and elemental components determination. This test was designed to achieve two objectives. First, to check if the tested chemical composition of efflorescence on the hydrated sample is in agreement with the previous XRD findings. Second, to compare its consistency with the efflorescence front (white deposits line) precipitated on carbonated paver has the same chemical compounds as the efflorescence on the hydrated pavers, found to form just above the water suspension line during wicking. The electron micrograph images of each sample and the corresponding EDX spectra for several specific points are shown in Fig. 4-22 to 4-25.

Figure 4.22 is taken for the efflorescence generated on the hydrated pavers. It reveals a dense mineral stacking morphology with evident platelet-like crystals. The EDX for the targeted points are shown in Figure 4.24, and identify these platelets as consisting of potassium, sulfur and oxygen, which complements results obtained from XRD, thereby confirming that the efflorescence was indeed a potassium sulfate compound.

While a little opaque, due to challenges in beam focusing, the morphology of the efflorescence formed in the carbonated pavers reveals monoclinic-type crystals (Figure 4.23). As shown in Figure 4.25, EDX generally gave the same elemental identification of potassium, sulfur, and oxygen as the three highest relative percentages, but identified a much higher oxygen peak and a more pronounced presence of sodium. This may suggest that the formed crystals share the same general compositional features of the hydrated efflorescence, as being a potassium-sulfate-

type compound, with a slightly more complex mineral makeup. Therefore, efflorescence observed in both the hydrated and carbonated pavers appears to generally have similar chemical features, but different morphologies and different locations of appearance within the pavers.

Findings from SEM and XRD are largely in agreement in that efflorescence observed on both the hydrated and carbonated pavers is generally potassium-sulfate-based. This is evidence that carbonation curing did weaken pore solution migration, refraining wicking from reaching to the service surface, and, therefore, the dissolved potassium sulfate alternately precipitated on the sides. Since efflorescence is primarily an aesthetic defect, carbonation curing presents a practical solution to this problem as long as efflorescence can be controlled under the visible surface.

The source of this sulfate-based efflorescence is very likely from the gypsum, anhydrite, and hemihydrate components of cement, which are normally added to control flash setting (Chin and Petry 1993, Brocken and Nijland 2004). The potassium salt can be an impurity found either in cement or the aggregate material.



Figure 4-22: Hydrated top efflorescence SEM



Figure 4-23: Carbonated bottom efflorescence Front SEM



Figure 4-24: EDX for hydrated paver efflorescence



Figure 4-25: EDX for Carbonate Paver Efflorescence Front

## **Chapter 5 Conclusion and Recommendations**

## **5.1 Conclusion**

The modified wicking test has proven to be an effective method to evaluate the potential of accelerated efflorescence for concrete pavers. Compared to ASTM C67, several modifications were made and summarized as follow: Stoppers were used to lift the sample and increase the water exposure surface, container size was minimized, and the air movement speed on the surface shall be increased to enhance the evaporation driving force. A considerable of efflorescence was prompted after the 10-day test.

In order to avoid human subjectivity intervention, instead of using the examination method introduced in the ASTM C67, an image analysis technique was devised consisting of image acquisition and a MATLAB image processing. Image analysis was relatively accurately in quantifying the effloresced area, and this technique was used throughout the project to evaluate the severity of efflorescence.

Early age pure CO<sub>2</sub> carbonation curing successfully eliminated the formation of efflorescence on the top exposure surface of the pavers compared to the otherwise heavily contaminated hydrated pavers, after both batches underwent the modified wicking test. Carbonation curing seems to prevent the pore solution from migrating up to the service surface. Instead, a line of white deposits precipitated on the sides of the pavers just above the water submersion line. Since efflorescence is mainly considered an aesthetic concern, pure CO<sub>2</sub> carbonation curing was shown feasible in keeping efflorescence out of sight. Flue gas curing with 20% CO<sub>2</sub> concentration slightly reduced efflorescence intensity, but salt precipitation was still visible on the pavers' top surfaces. Lower absorption and calcium hydroxide consumption seem to be the main features associated with pure  $CO_2$  carbonation curing and ones to contain efflorescence. It is well known that the carbonation curing is a CH consumption process, and CH is the essential chemical component causing carbonate based efflorescence. With the penetration of carbon dioxide during the curing process, more and more calcium carbonate fills the pores, and resulting in decrease in permeability, porosity and absorption. Semi and fully immersed absorption test were conducted to prove that the absorption and permeable voids volume of carbonated paver were lower than that of hydrated reference. And the water migration in the concrete is the leading cause of degradation (Hycrete 2011). Therefore, the carbonated concrete has a better general resistance to the weathering.

From X-Ray Diffraction and Scanning Electron Microscope (SEM) coupled with Energydispersive X-ray spectroscopy, the crystalline deposit of salts formed on the hydrated samples was identified as potassium sulfate. Sulfate may originally come from control setting mixtures in cement like gypsum, anhydrite and hemihydrate. And the potassium is probability contributed by the small amount of potassium existed in the cement and granite.

Moreover, the carbonation curing was tested that it has the capability to enhance the early compressive strength of the paver, and this technique gives more flexibility for manufactures to store and transport the products and reduce production cycle at the same time.

In principle, early age carbonation curing is able to prevent all kinds of efflorescence regardless of the chemical compositions and sources, since all of those types have the same forming mechanism. Carbonation curing provides a new efflorescence prevention solution for the concrete industry. Unlike the existing removal and prevention methods, this technique can eliminate the salt deposits on the visible service surface without damage or alter the desired texture and color. Furthermore, manufactures could benefit from the higher early age strength to reduce the production cycle and it is easier for them to manage storage and transportation. In addition, carbonation curing is an environmental friendly process, large amount of carbon dioxide will be consumed if the technique can be adopted by the industry.

### **5.2 Recommendation**

Further investigation and studies are still necessary, and the following list identifies some of the major items:

- Investigation into the sources of potassium and sulfate precipitated on the surface of hydrated pavers. The sulfate might come from the control setting additives, and the potassium seems originally from the cement or the granite.
- 2. In this research, the concrete paver itself was the only source to provide soluble salts. But it is not true in reality, use paver as an example, the paver needs to be installed on the bedding sand and also surrounded by joint sand. And also, the water is no longer deionized. Therefore, more severe test needs to be performed, including all the possibility source of soluble salts into account.
- 3. Flue gas carbonation curing failed to prevent the efflorescence completely in this project. Because of the economical advantage, further investigation can focus on using gas with a higher concentration of carbon dioxide to cure the sample. And the CO<sub>2</sub> uptake will increase consequently, a better capability of eliminating efflorescence supposed to be observed.
- 4. The pressure during carbonation curing was raised up to 5 bar, in order to get a higher CO<sub>2</sub> uptake and a lower permeability. Additional tests should be conducted to find the most cost-effective pressure can prevent the efflorescence by changing the carbonation curing conditions.

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