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Pore Size Distribution of Mortar in Near Surface Concrete Its Measurement and Delineation with Carbonation

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ABSTRACT

Ingress of deleterious agents from the environment causes deterioration of concrete and loss of its durability. Since the ingress takes place through the surface, characteristics of the near surface concrete has become an important issue in recent years. The permeation characteristics of near surface concrete are greatly responsible for concrete durability. Tests available for assessment of permeation characteristics such as Initial Surface Absorption, Figg Permeation and Covercrete Absorption are discussed in the introductory part of this paper. The pore size distribution of the mortar in the near surface concrete is considered an important parameter governing the durability of the concrete. Simultaneous measurement of pore size distribution by mercury porosimetry and carbonation rate by phenolphthalein test was made on samples exposed outside in the tropical marine environment of Singapore. The carbonation rate is then delineated with pore size distribution. Results of this pore size distribution and carbonation rate measurements in near surface mortars of several concretes are reported in this paper.

1.0 INTRODUCTION

1.1 GENERAL

The deterioration of concrete usually involves movement of aggressive gases and/or liquids from the surrounding environment into concrete, followed by physical and/or chemical actions within its internal structure possibly leading to irreversible damage (Basheer et al. 1993). Hence, the surface and near surface permeation characteristics, rather than the mechanical properties are the important factors for concrete durability.

The permeation characteristics can be assessed using tests such as Initial Surface Absorption (ISAT) (Levitt, 1969), Figg Permeation Methods (Figg, 1972) and Cover concrete (Covercrete) Absorption test (CAT) (Dhir, 1987).

Dhir et al (1989) showed that the potential depth of carbonation may be predicted from the Figg Air Index. Moreover, Dhir et al (1991) demonstrated that even mechanical properties such as the abrasion resistance of concrete correlated to the ISAT results as both tests assess the quality of cover concrete.

1.2 INITIAL SURFACE ABSORPTION TEST (ISAT)

ISAT was developed by Levitt (1969) as a quality control test for precast concrete tiles and kerbs. It is described in detail in BS 1881: Part 5: 1970. The revised version issued in 1983 (BS 1881: Part 122) has only minor changes.

The ISAT apparatus is shown in figure 1. It consists of a U-tube type structure with an opening at the bottom in the form of a circular cap (diameter 90mm) with surface area greater than 5000 mm^2. The cap is capable of being sealed to the concrete surface to withstand a small pressure of 202 N/m^2 (200mm head of water) by means of a clamp. Chan (1986) modified the ISAT apparatus to make it more compact, portable and easier to use. Dhir et al (1987) used a 'model air craft engine elastic' similar to an elastic band to provide better seal for the cap on the concrete surface. The ISAT cap was further modified by Byars (1991) by introducing a slope to the roof in order to flush the air bubbles rapidly prior to testing. One of the open ends of the U-tube is fitted with a reservoir which can be isolated from the system. The other is attached to a calibrated capillary tube supported horizontally by a frame 200mm above the concrete surface. The system is filled with water expelling any air bubbles. The amount of water absorbed by the concrete surface per minute (ISA value) is measured at 10, 30, 60 and 120 minutes.

1.3 FIGG AIR PERMEATION INDEX (API)

Figg (1973) described the development of a test for air and water permeability of the cover concrete. Cather et al (1984) modified the test technique in order to make it more applicable on site. Dhir et al (1987) reported a problem in using the Figg water test due to possible inconsistency in the manual injection of water in concrete. It was concluded that the test is unreliable.

In the same publication, the Figg Air Test was modified to give more repeatable results.

In the API test apparatus a hole of ⌀ 30 x 50 mm is drilled in the concrete surface. A polythene disc bridge and silicon rubber are inserted to block off the top 20 mm of the hole. During the test air is evacuated from the test cavity using a hypodermic needle connected to a hand vacuum pump (55 KPA below atmospheric pressure). The elapsed time for the pressure to increase from 55 to 45 KPA below atmospheric pressure is considered an index of the air permeability of concrete.

1.4 COVER CONCRETE "COVERCRETE" ABSORPTION TEST (CAT)

Dhir et al (1987) developed CAT in order to test the first 50 mm of the concrete cover to the reinforcement. A hole similar to that drilled in the API is used. A basic ISAT set-up is used but with a 13 mm internal diameter cap. An inlet tube located inside the sealed hole is used to supply water to the hole as shown in figure 2. The procedure for CAT is similar to that for ISAT.

1.5 APPLICATION OF THE PREMEATION TESTS IN SITU

The effect of the moisture content of concrete on the permeation test results is considerable. For example a concrete sample with a strength of 30 N/mm^2 when tested after water soaking for 2 days gave an ISA value of 0.25ml/m^2/s. This is classified as low sorptivity concrete in accordance with the concrete society recommendations (1986).

If, however, the same sample is tested after a long period of air drying, the measured ISA value was 50 ml/m^2/s which is classified as high sorptivity (Dhir et al 1987). Despite this, there is no standard preconditioning method which is suitable for both laboratory and in situ applications. BS 1881: parts: 1970 recommends two days air drying as a preconditioning method for in situ concrete. The obvious variations in exposure to sun, wind and rain makes this approach unviable. Dhir et al (1993) presented a technique for preconditioning concrete prior to the application of ISAT. The technique is based on drying the concrete surface by withdrawing moisture using a vacuum pump. The vacuum is applied to a modified ISAT cap containing silica gel granules which turn from pink to blue as the drying process proceeds. After drying is completed, the ISAT may be carried out in situ by fixing the cap to the structural member using suction cups. The measurements must be taken within 10 minutes from the release of vacuum pilot. In situ trials have shown that the test procedures reproducible results, is non destructive and simple to operate.

Shaaban and El Sayad (1994) applied the same vacuum technique to precondition samples for the API and CAT tests. It was reported that applying the vacuum directly to the hypodermic needle or the cap in API and CAT respectively, leads to better reproducibility in the results. However, the vacuum technique was found to be less sensitive with API and CAT than with ISAT. The authors attributed this to the nature of the former tests which introduce drilled holes in concrete and therefore the heterogeneity of the cavity may affect the measurements.

1.6 PRESENT STUDY

The quality of near surface concrete differs widely on the moulded (concrete in contact with formwork) and the unmoulded (open surface of concrete) surfaces. Pore size distributions of mortar in near surface concrete and the carbonation depths at the moulded and the unmoulded surfaces of various concretes exposed for two years in the tropical environment of Singapore are reported in this paper.

2.0 EXPERIMENTAL DETAILS

Concrete panels of varying grades were cast and cured in water for 28 days. The panels were then exposed for 2 years at two sites in Singapore, namely (i) a rooftop at the National University of Singapore (NUS) which is an inland location, and (ii) the East Coast Park which was bordering on the ocean. The concrete panels numbered 1, 2, 3, 5, 8, 9, 10 and 11 were exposed at the East Coast Park, whereas panels numbered 3, 4, 6, 7, 12 and 13 were exposed at the NUS roof top.

After the 2 years exposure, the panels were returned to the laboratory and the carbonation depths measured at both the moulded surface and the unmoulded surface. The phenolphthalein test was used to determine the carbonation depth. The phenolphthalein test is the simplest test among all the tests available for measuring carbonation depth. It also gives immediate indication and is an indication of the useful life remaining in existing structures. Phenolphthalein is a colourless acid/base indicator which turns purple when the pH is above a value in the range of 8.4 to 9.8, that is, when the concrete is alkaline. The phenolphthalein is prepared as a 1% solution in 70% ethyl alcohol. The solution is sprayed onto a freshly broken surface which has been cleaned of dust and loose particles. The measurement is carried out immediately after the broken surface has been exposed, alkaline areas of concrete turning a vivid purple colour. If no coloration occurs, carbonation has taken place and thus the depth of the carbonated surface layer can be measured. The main limitation of this test is that the procedure will cause localised surface damage and this method provides only an indication of the extent of carbonation.

Mortar pieces were extracted from the two regions of each concrete panel for pore size distribution measurements using a mercury pososimeter (Micromeritics Poresizer 9320) which covered the pore diameter range from approximately 360 to 0.006 μm. It should be emphasised that it was only the mortar that was used for the porosity determinations.

The poresizer measures the volume distribution of pores in materials by mercury intrusion or extrusion. Mercury porosimetry is based on capillary law governing liquid penetration into small pores. This law, in the case of non-wetting liquid like mercury and cylindrical pores is expressed by the Washburn [1921] equation:

$$d = \frac{-4Y \cos\theta}{P}$$

where d = equivalent diameter of the intruded pores
Y = surface tension of mercury
θ = angle of contact between the mercury and the pore walls
P = pressure at which a given incremental of mercury intrudes into the pore system

The volume of mercury, V, penetrating pores is measured directly as a function of applied pressure and this serves as a unique characterization of pore pressure. Pores are rarely cylindrical, hence the above equation constitutes a special model which may not best represent pores in actual materials. However its use is generally accepted as the practical means for treating what, otherwise would be a most complex problem.

In these studies, a sample to be tested is first broken into smaller pieces and then dried in an oven for at least 24 hours. When cooled, the specimen is then put into the penetrometer to be inserted into one of the pressure ports. As mercury is non-wetting to most materials, it will not penetrate into the pores without hydrostatic pressure. These properties cause a mercury surface in contact with a solid to assume the minimum surface area and largest radius of curvature possible at a given pressure. An increase in pressure on the mercury shifts the balance between surface tension and surface area causing the radius of curvature of the mercury contacting the solid to become smaller. When the radius is equal to that of a pore entrance, mercury fills the volume within the pore. Thus after evacuating the sample, pressure is applied to force the mercury into the pores of the sample. The volume of mercury penetrating the pores was measured directly as a function of applied pressure. As the pressure increases, mercury intrudes into smaller and smaller pores. The samples are put under low and high pressure to ensure full intrusion of mercury. Once the intrusion of the mercury has been completed, the pressure is released to extrude the mercury from the pores.

The surface tension of the mercury was taken as 0.485 N.m^{-1} and the advancing contact angle is assumed to be 130.0^{0} [1993]. Upon completion of the process, the following parameters were determined:

i) total intrusion volume
ii) total pore area
iii) median pore diameter
iv) average pore diameter
v) apparent and bulk density

In the results section we present only the results for median pore diameter

3.0 RESULTS AND DISCUSSION

Figure 3(a) shows the results of the median diameter for both the unmoulded and moulded samples. Figure 3(b) shows the respective carbonation depths of both the unmoulded layers and moulded layers for the same 13 samples. Figure 3(a) shows that

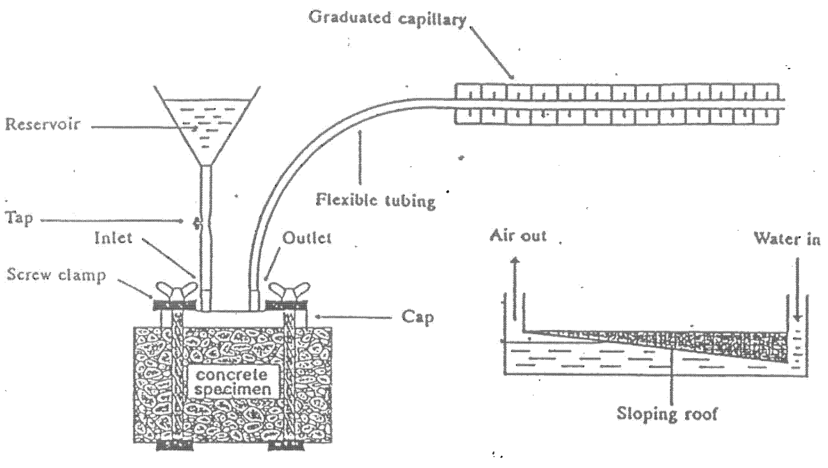

Fig.1 General layout of the ISAT equipment and sloping roof cap.

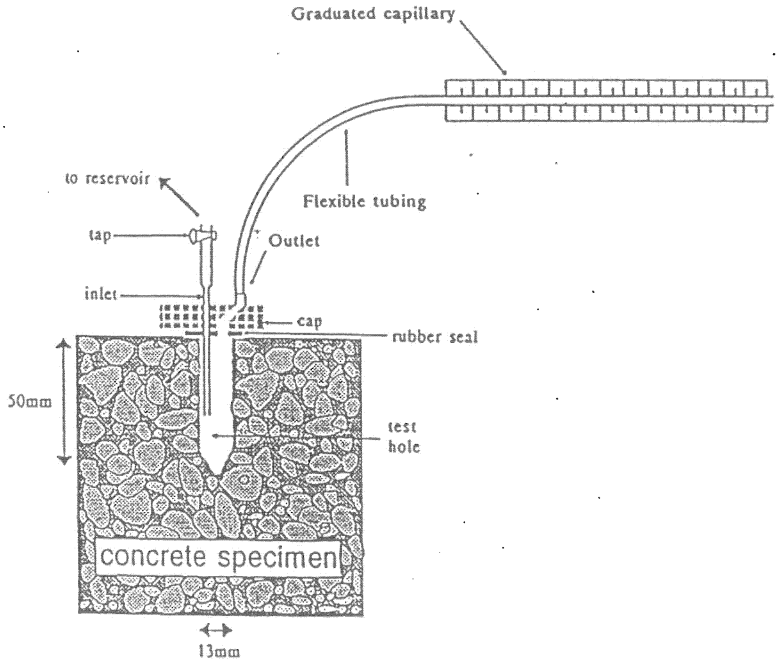

Fig.2 Schematic arrangement of CAT

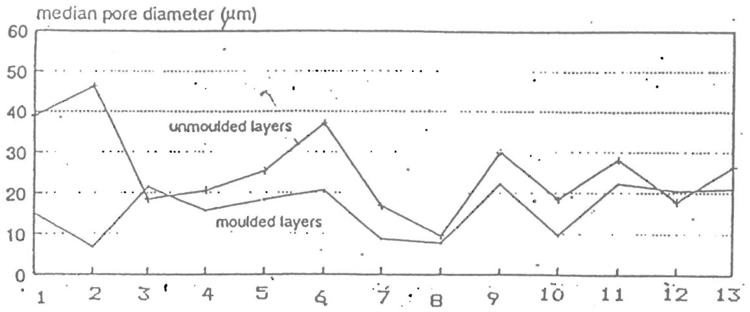

Fig.3(a) Median pore diameter vs concrete samples (moulded and unmoulded layers)

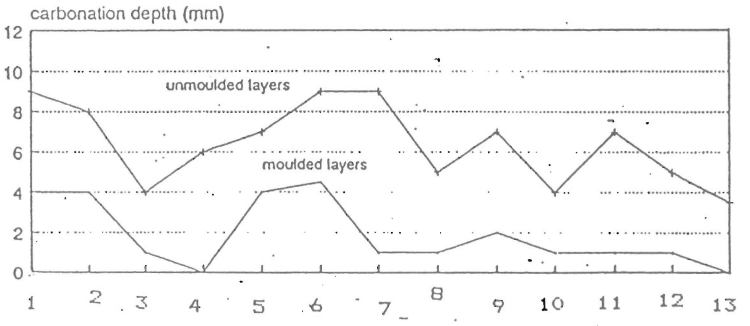

Fig.3(b) Carbonation depth vs concrete samples (moulded and unmoulded layers) for 11 of the 13 samples, the median pore diameters for the unmoulded samples (with larger carbonation depth) are greater than those of the moulded samples (smaller carbonation depth). The results shown in the two figures confirm that the unmoulded regions, where larger carbonation depths were found, have larger pore size as well. Figure 3(a), it can be seen that the median pore diameter of the moulded layers of two samples, are slightly greater than that of the unmoulded layers. This could be due to the fact that the pores of the carbonated concrete have been reduced as a result of the formation of some calcium carbonate. On the other hand, for the 11 samples whereby the median pore diameter of the unmoulded concrete is greater than the moulded concrete, the calcium carbonate produced in the largely carbonated unmoulded concrete may not be large enough to result in a median pore diameter smaller than that in the less carbonated moulded concrete. It is suggested that a petrographic examination be done in conjunction with the mercury porosimetry test to confirm this hypothesis.

At a given humidity pores of a specific size are filled with water. In a completely dry concrete there is no water in the pores and hence carbon dioxide cannot react. If a concrete is completely saturated, the carbon dioxide has to dissolve in the pore water, diffuse through the pores, reach the concrete beyond the carbonation front, and then react; this also is a slow process. As a result, when pores are not saturated but have only a layer of moisture on their walls, carbon dioxide can rapidly diffuse through the pores and react on the moist pore wall surfaces. This explains how pore size of mortar in near surface concrete can influence carbonation under a given atmospheric condition.

4.0 CONCLUSIONS

1. Pore sizes in the mortars of the moulded and the unmoulded surfaces of a concrete are different. Unmoulded region have larger pores.

2. Carbonation rates of the moulded and the unmoulded surface, of a concrete are different. Unmoulded regions have higher carbonation depths.

3. Higher carbonation rate concrete is associated with larger pores of the mortar in the concrete.

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