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Feasibility of utilizing oven-drying test to estimate the durability performance of concrete

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Abstract. The increasing concern for reinforced concrete structure durability has been justifying in many ways in the last few decades. However, there is no perfect durability test method till now. In this research an alternative method, which is based on the cumulative moisture loss percent of the concrete specimens after oven-drying, was proposed to estimate the durability performance of the concrete. Two temperatures were considered for the oven-drying tests: 100°C and 200°C. Test results showed that oven-drying at 200°C was obviously an unsuitable procedure to preserve the fragile microstructure of cement-based materials. By contrast, experimental results through oven-drying at 100°C allowed estimating the moisture loss percent of cement-based materials in a more rational manner. Moreover, the magnitudes of the cumulative moisture loss percent obtained from oven-drying tests at 100°C for 48 hours have good correlations with the data of other well-known methods, namely, electrical resistance test, water permeability test, and mercury intrusion porosimetry test. This investigation established that regarding the oven-drying test as one of the tests for evaluating the potential durability of concrete is considerably practicable.

Keywords: concrete; durability; drying.

1. Introduction

Concrete is a man-made construction material that consists of cement, aggregate, water, and admixtures. During hydration and hardening, concrete needs to develop certain physical and chemical properties. Among other qualities, mechanical strength and durability (such as low moisture permeability, and chemical and volumetric stability) are necessary. In concrete, however, strength and durability are not necessarily linear to each other, more so when high grade cement is used. In other words, strength alone cannot ensure durability, which means durability is an independent factor to strength. Especially, the increasing concern for reinforced concrete structure durability has been justifying in many ways in the last few decades (Chen *et al.* 2008, Chen *et al.* 2009). For instance, the ACI 318 Building Code has emphasized the importance of durability at the design stage by having all durability requirements in one stand-alone chapter since 1989. Further, for the 2008 edition of Building Code Requirements for Structural Concrete, ACI Committee 318 (2008) has proposed a significant restructuring of the durability chapter. Accordingly, numerous project specifications in the world are increasingly including requirements for performing durability-

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related tests on hardened concrete. These suggest that concrete durability has been regarded as one of the most important considerations in the design of new reinforced concrete structures.

Unlike compressive strength, which is a definitive property, durability is not easily measurable. This is further complicated by the inherent unreliability of some of the test methods (Pocock and Corrans 2007). Nevertheless, the ability of concrete to withstand the penetration of liquid and oxygen can be described as the durability of concrete. Therefore, current test methods for assessing the durability of concrete are inferred primarily by measuring the resistance of the cover layer to transport mechanisms such as permeation, absorption, and diffusion (De Beer *et al.* 2005). In fact, it is well known that water in hardened cement paste can be presented as evaporable water in gel pores and capillary pores, as well as in cement hydrates as chemically bound water (Bazant and Kaplan 1996). When exposed to high temperature, concrete undergoes changes in its chemical composition, physical structure and water content. These changes occur mainly in the hardened cement paste and cause many intricate consequences (Tang 2010). Hilsdorf pointed out that at temperature less than 105°C, it can be assumed that the amount of chemically bound water is not affected by temperature (Hilsdorf 1967). However, gel and capillary water decrease parabolically at temperatures between 40°C and 105°C. By the time the temperature reaches about 100-200°C, water vapor pressure continuously forms in a zone close to the heated surface (Schneider 1985, Harmathy 1970). It results substantially from the rapid evaporation of water inside the concrete, when it reached and passes through the boiling point of water.

The moisture content of concrete under particular circumstance may affect its properties. Theoretically, at elevated temperatures the amount of all evaporable water expelled from the hardened cement paste can be associated with concrete durability. Therefore, an alternative method that is based on the moisture (or weight) loss percent of the concrete specimen after oven-drying tests was proposed to estimate the durability performance of concrete. In addition, this study made an effort to relate the evaporable water in concrete with the durability in the aspects of electrical resistance, permeability ratio, and total intruded pore volume. Accordingly, the comparisons with the frequently used test methods (such as electrical resistance test, water permeability test, and mercury intrusion porosimetry test) for concrete durability are also made to examine the feasibility of the presented approach.

2. Experimental details

2.1 Materials

Materials used here for making specimens included cement, fine aggregate, coarse aggregate, and superplasticizer. The cement was Type I Portland cement. The chemical composition and physical properties of the cement are shown in Table 1. The coarse aggregate was crushed limestone with a maximum size of 19 mm and the fine aggregate was natural river sand with a Fineness Modulus of 2.82. A superplasticizer conforming to ASTM C-494 Type G was used.

2.2 Concrete mixtures and specimens

To cover normal- and high-strength ranges, four values of the nominal 28-day concrete strength were chosen equal to 20, 30, 40, and 50 MPa. The mix proportions for the four concrete series are given in Table 2. For example, the M20 concrete mix with a water-to-cement ratio (W/C) of 0.75 was

Table 1 Chemical composition and physical properties of cement

Item	Cement
SiO_2, %	20.90
Fe_2O_3, %	3.21
Al_2O_3, %	5.65
CaO, %	63.63
MgO, %	2.52
SO_3, %	2.16
Na_2O, %	0.10
K_2O, %	0.52
C_3S, %	48.76
C_2S, %	23.41
C_3A, %	9.54
C_4AF, %	9.77
Loss on ignition, %	0.92
Specific gravity	3.15
Specific surface area, m^2/kg	344

designed for a specified compressive strength of 20 MPa at 28 days. The mixing continued until a uniform concrete without any segregation was obtained. Along with each mixture, enough 150 mm diameter × 300 mm high cylindrical specimens were cast. Following casting, all the specimens were covered with a wet hessian. Then, the cylinders were removed from the molds 24 hours after casting. All specimens were cured in a saturated calcium hydroxide solution bath at 23 ± 2°C for 27 days. The slumps of fresh concrete were measured according to ASTM C143. As for the 28-day compressive strength of hardened concrete, it was measured according to ASTM C39. The results of compressive strength are reported in Table 2, in which each value was the average of three specimens for each mix proportion at 28 days.

2.3 Tests

Most durability tests are essentially intended as a measure of the pore structure of concrete, but

Table 2 Mix proportion of concrete and test results

Mix No.	W/C	Quantities, kg/m^3					Test results	
		Water	Cement	Coarse aggregate	Fine aggregate	SP	Slump, cm	f_c', MPa
M20	0.75	190	253	1110	801	2.57	16.5	22.9
M30	0.60	190	317	1110	737	3.53	16.0	36.3
M40	0.50	190	380	1110	674	4.62	15.0	46.7
M50	0.45	190	422	1110	632	6.00	17.0	51.8

Note: W/C=Water-to-cement ratio; SP=Superplasticizer; f_c'=Compressive strength.

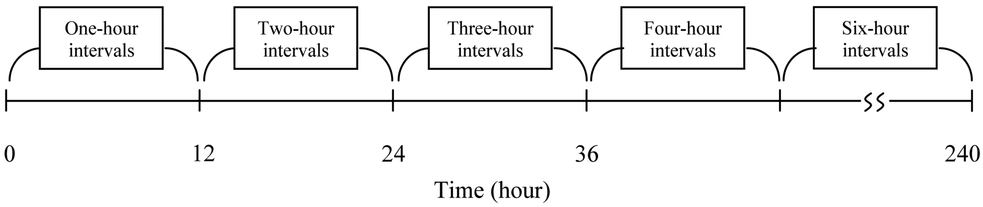


Fig. 1 Time intervals for measuring specimen's weight

the pore structures of different concretes develop at different rates. Therefore, tests are usually performed on standard laboratory test specimens at 28 days. In view of this, the specimens were cured in water for 27 days before being dried in a computer-control electric furnace. Two temperatures were considered for the oven-drying tests: 100°C and 200°C. In the study the rate of heating was 5°C/minute until the temperature inside the furnace reached the target temperature. Then the specimens were taken out and weighed at specific time intervals to obtain the amount of moisture loss during these intervals (See Fig. 1). The moisture loss percent, W_L, is defined by the following expression

$$W_L = \frac{W_i - W_{i+1}}{W_s} \times 100\% \quad (1)$$

where W_s = the saturated-surface-dry weight of specimen before oven-drying test (g); W_i and W_{i+1} = the weight of specimen after drying in an electric furnace for i and i+1 hours (g), respectively. The cumulative moisture loss percent, W_A, and the total moisture loss percent, W_T, of the specimen are calculated from the following expressions respectively

$$W_A = \frac{W_s - W_i}{W_s} \times 100\% \quad (2)$$

$$W_T = \frac{W_s - W_c}{W_s} \times 100\% \quad (3)$$

where W_c = the weight of specimen after drying in an electric furnace to a constant weight (g).

Water permeability test was conducted on the oven-dry specimens that had been dried in an electric furnace until constant moisture loss was reached. The test was a pressure induced permeability test. The specimen was positioned into the apparatus and subjected to a constant pressure head. At one end of the specimen, water was introduced with a constant driving pressure of 0.98 MPa for three hours. Then the specimen was weighed to obtain its wet weight. The difference between the wet weight and the oven-dry weight of the specimen is the weight of water that penetrated into the pore structure of the concrete. The following equation is used to calculate the permeability ratio, P_p, of the specimens

$$P_p = \frac{(W_P - W_c)}{V_V \cdot \gamma_w} \quad (4)$$

where W_P = the weight of specimen after water permeability test (g); γ_w = the density of the water (g/cm^3); and V_V = the volume of the specimen (cm^3).

The porosity and pore size distribution was measured by using a mercury intrusion porosimetry.

The 5 mm thick samples used were obtained from identically produced and cured specimens at 28 days. The fragments were dipped into methanol for 5 minutes to stop the hydration, and then dried in the vacuum desiccators for 2 days. The pressure of mercury intrusion porosimeter ranges from 1.4 MPa to 414 MPa. The pressure required is a function of the pore size and can be converted to equivalent pore width using the Washburn equation, as in Eq. (5) (Feldman and Beaudoin 1991, Cook and Hover 1999, Galle 2001)

$$D = \frac{-4\gamma\cos\theta}{P} \tag{5}$$

where D = the equivalent pore width (m); P = the absolute pressure exerted (Pa); γ = the surface tension of mercury (N/m); and θ = the contact angle (wetting angle) between solid and mercury (^{o}). The relationship is commonly known as the Washburn equation. A surface tension of 480 mN/m and a contact angle of 140^o were used in the Washburn equation to convert applied pressure to pore diameter.

Electrical resistivity was measured by a Surface Resistivity meter with a Wenner linear four-probe array that consist of four equally spaced electrodes connected to a source of alternating current, and the inner electrodes are connected to a voltmeter. For resistance measurements, the four electrodes should be touching the concrete surface. The apparent resistivity, ρ (unit: Ohm-cm), is given by the following expression (Polder 2001, Chung 2002)

$$\rho = \frac{2\pi a V}{I} \tag{6}$$

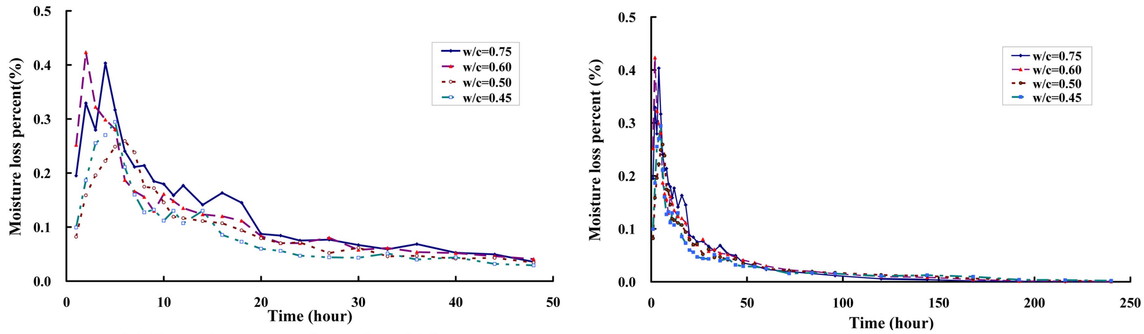
where a = the probe spacing (cm); V = the measured voltage between the inner electrodes (Volt); and I = the current between the outer electrodes (Ampere).

3. Results and discussion

3.1 Oven-drying tests

As previously mentioned, the evaporable water in a hardened cement paste comprises water held in the capillary pores and absorbed water in the gel pores. This amount can be determined by oven drying a sample at a temperature of 105°C. By contrast, chemically bound water is a measure involving the microstructure of the hydration product and is obtained from a paste heated to very high temperatures (1000°C). Accordingly, in the study it is assumed that at temperature less than 105°C the amount of the chemically bound water is not affected by temperature.

Fig. 2 shows the experimental moisture loss percents (as a percentage of the initial weight at room temperature) versus duration of heating at 100°C for specimens with various W/C, in which each value of the moisture loss percent is the average of three concrete cylinders for each mix proportion at 28 days. It can be observed from Fig. 2(a) that the peak value of the moisture loss percent depended on the W/C ratio. In addition, the moisture loss percent was most rapid after about 2 to 6 hours heating and the magnitude of the moisture loss percent was in the range of 0.15% to 0.43%. Moreover, after oven-drying at 100°C for 48 hours, the moisture loss percent was not entirely stable. The reason is the drying of the cementitious phases is a continuous phenomenon and thus the duration of oven-drying must be prolonged to reach stable condition. In fact, Fig. 2(b) shows that,



(a) Duration range from 0 to 48 hours

(b) Duration range from 0 to 240 hours

Fig. 2 Plot of W_L vs. duration for oven-drying at 100°C

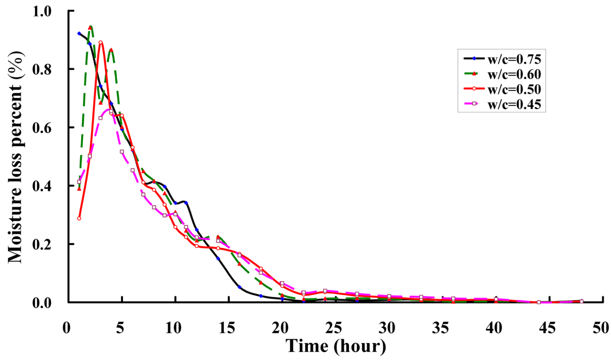
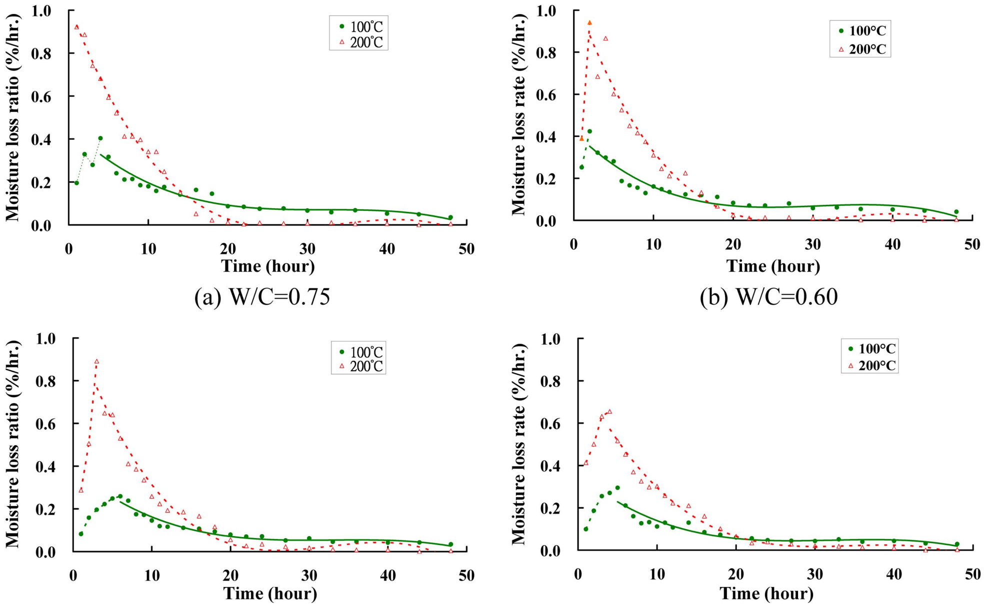


Fig. 3 Plot of W_L vs. duration for oven-drying at 200°C

after oven-drying at 100°C for a sufficient length of time (i.e. 240 hours), the moisture loss percent could be reduced to zero since all evaporable water had been expelled from the hardened cement paste and aggregate.

For specimens heated to 200°C the moisture loss percent was most rapid after about 1 to 4 hours heating and the magnitude of the moisture loss percent proceeded at an increased rate that was in the range of 0.36% to 0.97%, as shown in Fig. 3. In addition, Fig. 4 shows the plots of moisture loss ratio versus duration of heating (in the first 48 hours) at 100°C and 200°C for specimens with various W/C. It can be clearly seen in Fig. 4 that the moisture loss ratio increased in the first few hours of heating duration and then decreased to a stable value. Further, compared with Fig. 2(a), Fig. 3 reveals that after oven-drying at 200°C for 48 hours, the moisture loss percent was entirely stable. The reason for this tendency is that, at 200°C, free water was completely removed from porosity.

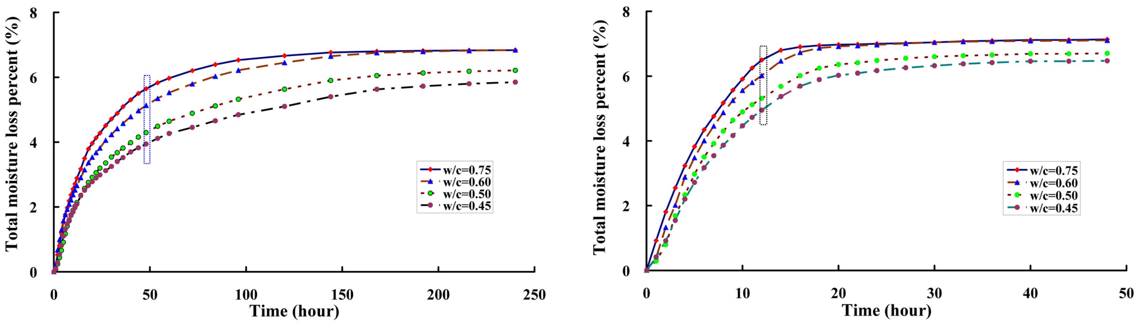
The total moisture loss percent of the concrete specimens obtained through 100°C and 200°C oven-drying are compared in Fig. 5. It can be observed that the total moisture loss percent of the concrete specimen approached a limit with the increase of the duration of oven-drying. Besides, total moisture loss percent increased with the increase of W/C ratio. This trend is ascribed to the higher amounts of capillary pores forming the more free water without participating the hydration of cement for the paste with high W/C ratio. In other words, the higher volume of internal pore structure was, the more easily the moisture released. And thus it will result in the higher total



(a) W/C=0.75 (b) W/C=0.60

(c) W/C=0.50 (d) W/C=0.45

Fig. 4 Plot of moisture loss ratio vs. duration



(a) Oven-drying at 100°C (b) Oven-drying at 200°C

Fig. 5 Plot of W_T vs. duration

moisture loss percents and be harmful to the durability of concrete. On the other hand, it can be observed in Fig. 5 that after oven-drying at 100°C for 48 hours or at 200°C for 12 hours the differences among the cumulative moisture loss percents of the concrete specimens with various W/C ratios were much obvious. This will be discussed and validated with the results obtained with conventional tests later.

3.2 Comparisons with conventional tests

During the oven-drying test, the moisture content of concrete at elevated temperature decreased from an original value to the final equilibrium value at a decreasing rate. After 100°C and 200°C oven-drying tests, the conventional tests (i.e. electrical resistance test, water permeability test, and mercury intrusion porosimetry test) were conducted on the specimens to relate the evaporable water in concrete with the durability in the aspects of electrical resistance, permeability ratio, and total intruded pore volume. For the sake of consistency, the specimens used for oven-drying test were re-used for other tests to produce reliable values to be used for comparative purposes.

Fig. 6 shows the data of permeability ratios for the specimens with various W/C values. It is clear

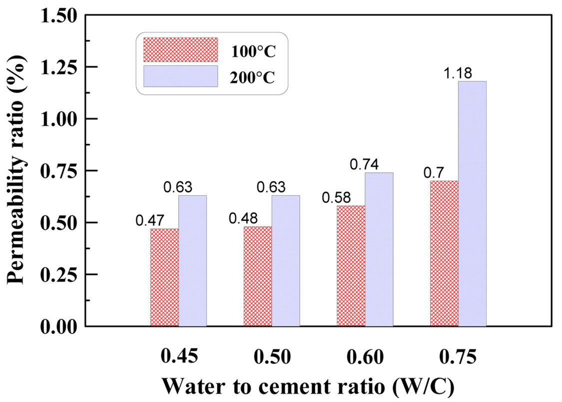


Fig. 6 Permeability ratio histograms for concretes with various W/C values

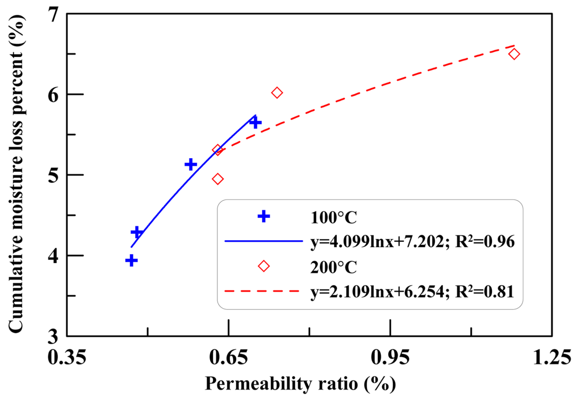


Fig. 7 Plot of cumulative moisture loss percent vs. permeability ratio

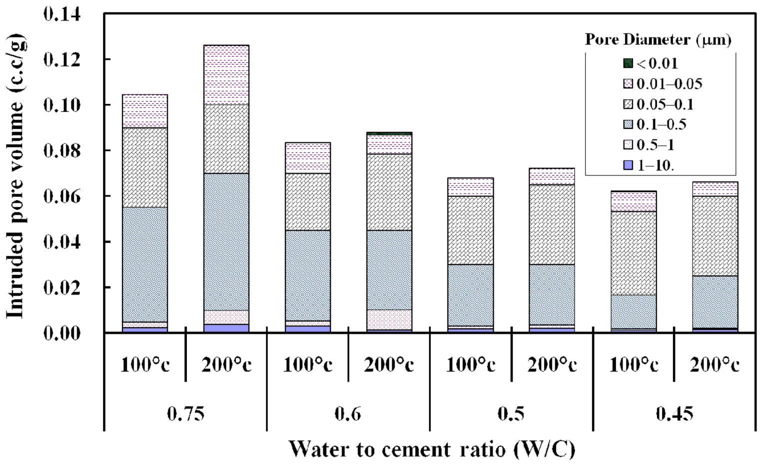


Fig. 8 Intruded pore volume histograms for concretes with various W/C

that W/C ratio had significant effect on permeability ratio. Furthermore, the values of permeability ratio obtained through 200°C oven-drying were obviously greater than those obtained through 100°C. Fig. 7 is the plot of cumulative moisture loss percent, obtained after oven-drying at 100°C for 48 hours and at 200°C for 12 hours, versus with permeability ratio. The correlation coefficients by nonlinear regression are 0.96 and 0.81 for oven-drying at 100°C for 48 hours and at 200°C for 12 hours, respectively.

Fig. 8 shows the intruded pore volume histograms and the pore-size distribution for concrete with various W/C ratios after 100°C and 200°C oven-drying tests, where the pore volume is based on the

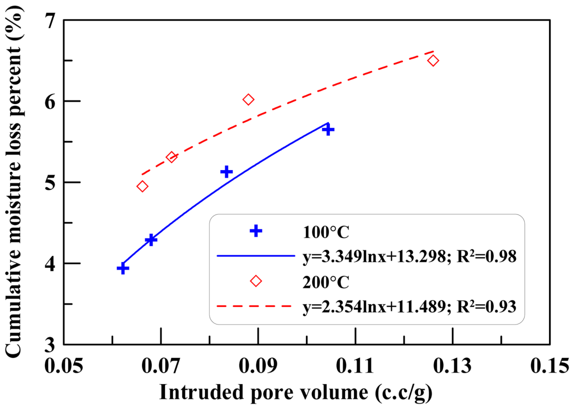


Fig. 9 Plot of cumulative moisture loss percent vs. total intruded pore volume

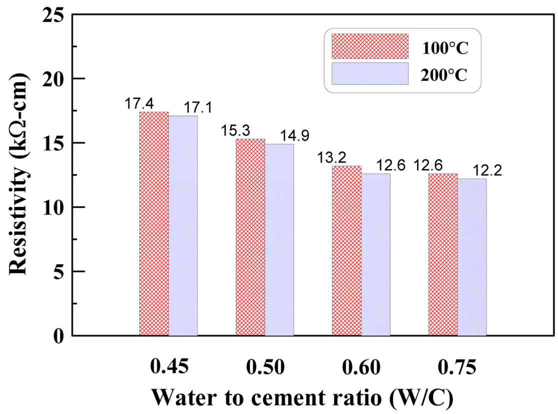


Fig. 10 Electrical resistivity histograms for concretes with various W/C

volume of the paste portion of the specimen. It can be observed that the concrete specimens treated at 100°C and 200°C were characterized by similar pore size distributions. But the final intruded pore volumes increase particularly for concrete with a W/C ratio of 0.75 when the specimens removed from oven-drying at 100°C to 200°C. The difference in pore volume is ascribed to the transformation of the fine pore structure into a coarser one due to dehydration of calcium silicate hydrates (C-S-H). This hints that oven-drying at 200°C generates important damages for the materials microstructure in the capillary porosity domain. In other words, oven-drying at 200°C leads to an overestimation of total porosity. Fig. 9 is the plot of cumulative moisture loss percent versus total intruded pore volume. The correlation coefficients for the nonlinear fit are 0.98 and 0.93 for oven-drying at 100°C for 48 hours and at 200°C for 12 hours, respectively.

After oven-drying test, concrete specimens were prepared in saturated surface dry condition. Then the electrical resistivity of the concrete specimens was measured. Fig. 10 shows that the resistivity increased with the decrease of W/C ratio, as expected. Moreover, the values of electrical resistivity obtained through 100°C oven-drying were greater than those obtained through 200°C. This behavior is because drying at 200°C led to damage of the pore structure of the hydrated cement paste in concrete specimens due to dehydration of C-S-H or microcracking and thus causing an increase of moisture content in pre-saturated specimens (Harmathy 1970). As a result, electrical resistance decreased as moisture content increased. Fig. 11 is the plot of cumulative moisture loss percent versus electrical resistivity. The relationships were approximately parallel to each other, and, in both cases, the correlation coefficients for the nonlinear fit were greater than 0.93, as represented in Fig. 11.

A number of standard test methods have been developed to test concrete resistance against specific deterioration mechanisms, e.g. salt surface scaling, water absorption, water permeability, and chloride ingress. However, at present there are no accurate and perfect durability test methods. Therefore, it is essential to consider the issues presented when assessing durability results, and not to view results in isolation, but rather to assess results from the various tests collectively (Pocock and Corrans 2007). The amount of evaporable water in the hydrated cement paste affects the durability of the hardened concrete. Especially, excessive water can exacerbate freeze-thaw damage and alkali-silica reactivity, and make the concrete more permeable. In general, oven-drying test is probably the most widely used technique to determine the moisture content of

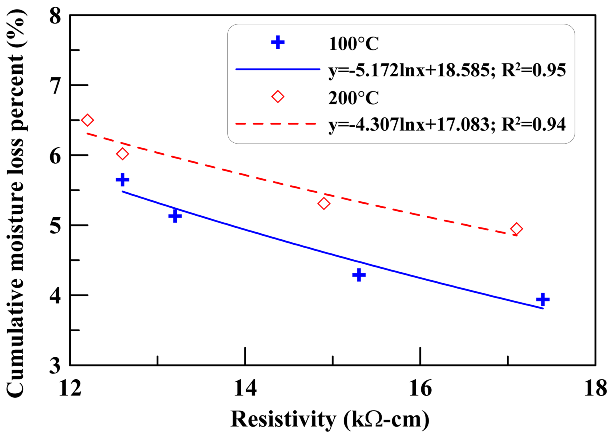


Fig. 11 Plot of cumulative moisture loss percent vs. resistivity

construction materials in civil engineering. In this research the water content was calculated by the weight loss of the concrete specimens after drying. The analytical results show that the higher the temperature, the greater was the rate and magnitude of moisture loss. But at the same time the amount of chemically bound water in the products of cement hydration was reduced. For example, C-S-H was partially dehydrated and thus pore structure in the hydrated cement paste was affected. Therefore it is considered that oven-drying at 100°C allows estimating the moisture loss percent of cement-based materials in a more rational manner although oven-drying at 200°C is a quicker drying procedure.

4. Conclusions

The above comparisons with conventional tests confirm that the magnitude of the cumulative moisture loss percent obtained from oven-drying tests at 100°C for 48 hours have good correlations with the data of standard concrete durability tests. Moreover, the use of oven-drying test has the advantage of being a relatively simple procedure that uses inexpensive equipment. These verify the applicability of this new technique for evaluating the potential durability of concrete.

On the other hand, it should be pointed out that the rate and magnitude of the moisture loss percent obtained from oven-drying test at a specific time of drying under specific conditions would be affected by factors such as the type of aggregate, the size and shape of the concrete specimens, the rate and period of heating, and environmental conditions. Further studies should be performed to investigate how these factors affect the correlation between the moisture loss percent and durability performance of the concrete.

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