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Evaluation of mass transfer resistance of concrete based on representative pore size of permeation resistance

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ABSTRACT

This paper shows that air and water permeability and water infiltration rate into concrete can be evaluated with the minimum pore size which mass has to pass through for penetration. The authors have proposed a new method to measure threshold pore size and reported that permeability of both air and water in concrete is mainly governed by threshold pore size. Based on the above findings, surface air permeability test is conducted on dried specimens with different thickness. Here, to avoid confusion with threshold pore size, representative pore size of permeation resistance (RPSPR) is defined as the smallest pore size which mass must pass through to penetrate a specimen. With water-sensitive paper, time for water penetration through concrete specimen with different thickness is measured and compared with an indicator calculated from RPSPR. They show very good correlation and this result indicates that RPSPR can be an indicator of water infiltration rate.

Keywords. Threshold Pore Size, Representative Pore Size of Permeation Resistance, Water Infiltration, Air Permeability, Water Permeability

INTRODUCTION

For quality verification of newly constructed concrete structures and efficient maintenance of existing concrete structures, more attention is paid to evaluation of concrete durability, more accurately. Considering the above purposes, the evaluation method should be non-destructive. So far, various methods have been proposed, however, all non-destructive method to evaluate mass transfer resistance of concrete gives us just qualitative information and it is impossible or not reliable to estimate mass transfer or deterioration rate of concrete members based on the obtained results. In the first place, in such measurements, it is not understood that what information, pore size distribution, total pore volume, etc., is measured. The authors have developed a method to extract threshold pore size, one of the indicators of pore structure, and confirmed that water permeability and air permeability, if the specimen is enough dry, are governed by threshold pore (Sakai, 2012). In this paper, first, it is proved that threshold pore size is extracted with the new method definitely. Next, based on the results of water penetration test, it is shown that representative pore size of permeation resistance (RPSPR) can be an indicator of water infiltration rate. Here, RPSPR is defined to avoid confusion with threshold pore size, as the smallest pore size which mass must pass through to penetrate a specimen. As the thickness of a specimen becomes less, there is more possibility for mass to penetrate it avoiding smaller pore and, as a result, RPSPR becomes larger. Threshold pore size is measured with MIP and is regarded as an indicator independent of specimen's thickness. As mentioned above, both threshold pore size and RPSPR are an indicator of pore structure which corresponds to the smallest pore size which mass must pass through to penetrate a specimen.

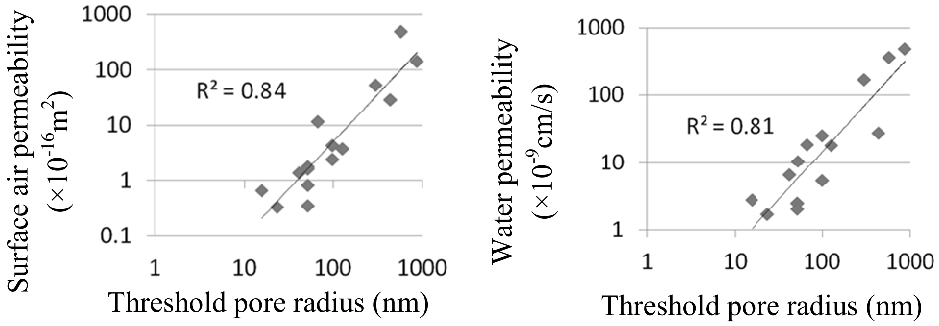
DISCUSSION ON THE RELATIONSHIP BETWEEN THRESHOLD PORE SIZE AND AIR/ WATER PERMEABILITY

So far, many researchers have pointed out that there is good correlation between threshold pore size and air and water permeability. Powers (1958) and Metha (1980) studied relationship between pore structures and water permeability. Powers found correlation between volume of capillary pore and water permeability, and Metha reported a good correlation between threshold pore size and water permeability. Here, threshold pore size is the minimum pore size which mass should pass to penetrate the objective, and pore size distribution is measured with Mercury Intrusion Porosimetry (MIP). Halamickova and Detwiler (1995) reported that there was a correlation between critical pore size, an indicator of pore structure, and both water permeability and coefficient of oxygen diffusion. Goto (1996) related threshold pore size with hydration rate of cement. The indicators of pore structures and permeability shown above, however, can't show such high correlation in various types of specimens since it is not easy to extract threshold pore size correctly, particularly, in samples taken from concrete specimens. The authors (Sakai et al., 2012) have proposed a new method to extract the threshold pore size by using epoxy-resin-coated specimen in MIP. Here, the definition of threshold pore size is following Winslow and Diamond (1970), the corresponding pore size where cumulative pore volume curve shows

Table 1 Mix proportion of concrete specimens prepared in experimental room

	W/B (%)	Curing condition	Unit : kg/m^3					Threshold pore (nm)
			W	C	FA or BFS	S	G	
N40-1	40	Water	180	450	-	708	978	52.5
N55-1	55	Water	180	327	-	805	984	52.5
N70-1	70	Water	180	257	-	886	960	52.7
FB55-1	55	Water	172	251	62	791	1007	15.7
FC55-1	55	Water	169	216	92	783	1017	42.2
BA55-1	55	Water	179	260	65	787	1002	126.7
BB55-1	55	Water	174	159	159	792	1008	99.3
N70-3	70	Wind	180	257	-	886	960	301.2
FB40-1	40	Water	172	345	86	694	998	23.7
FB70-3	70	Wind	172	197	49	873	985	866.9
BB40-1	40	Water	174	218	218	695	1001	52.4
BB70-3	70	Wind	174	124	124	873	985	577.4
L55-2	55	Sealed	180	327	-	807	987	437.5
M55-2	55	Sealed	180	327	-	807	987	67.3
H55-2	55	Sealed	180	327	-	804	984	99.3

the largest tangent. In our method, 5mm cubic piece of sample is coated with epoxy resin leaving a small area of around 4 mm^2, and analysed with MIP. The expected effect of coating is as follows; in normal sample, mercury tries to avoid smaller pores, including threshold pore, and, as a result, large part of the sample is already filled when mercury starts intruding into threshold pore. In this case, no sudden intrusion is observed at threshold pore size. On the other hand, when coating is applied to the sample, because of the limited open area, mercury can't avoid the smaller pore and less intrusion occur until it reaches enough pressure. In this case, sudden intrusion occurs at threshold pore size. The validity of the method was confirmed with concrete specimen of various mixing design and curing condition as shown in Table 1. AE water reducing agent and AE agent is used with the amount of 0.2% and 0.004% against cement weight, respectively. Specimens were demolded 24 hours after the casting and under-water, sealed, or in-wind curing were given until the age of 28 days, and after that, all specimens were cured in a room of 20 degree Celsius. Here, in in-wind curing, specimens are winded by a fan to accelerate drying. All tests were conducted when the age of the specimens were 2.75 years. It was confirmed that the threshold pore size (Table 1), obtained by the new method, and air and water permeability showed good correlation as shown in Fig. 1. The good correlation indicates that threshold pore size can be calculated from air or water permeability. In the next chapter, it is shown that sudden intrusion occurs at threshold pore size when the sample is coated with epoxy-resin.



(a) Surface air permeability and threshold pore radius

(b) Water permeability and threshold pore radius

Figure 1. Correlation between air/ water permeability and threshold pore size (Sakai et al., 2012)

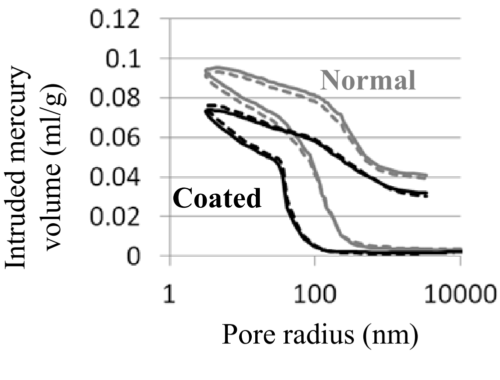


Figure 2. Comparison between with and without coating

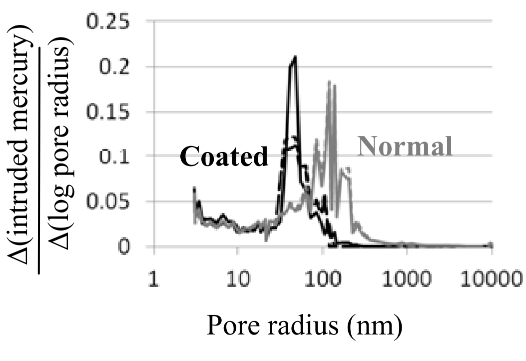


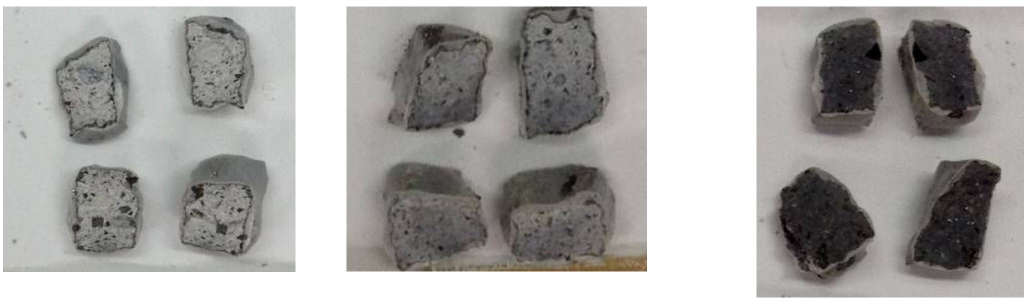
Figure 3. Tangent of Fig. 2

VERIFICATION OF MIP WITH EPOXY-COATED SPECIMEN TO EXTRACT THRESHOLD PORE SIZE

To confirm sudden intrusion in MIP at threshold pore size, when sample is coated with epoxy resin, additional study was done on mortar specimen. In the study, the splitting surfaces of the samples were observed after MIP analysis. Mortar of W/C=55%, C/S=30% was used. The specimens were demolded 24 hours after casting, cured under water until the age of 28 days, and dried in room of 20 degrees for half year. 5mm cubic pieces were taken from the mortar and immersed into acetone for 24 hours, dried by D-dry method, and measurement was executed on two pieces each case. Fig. 2 shows intruded mercury volume of samples with and without coating. Since each two curves show almost same behaviour, it can be said that this method has reproducibility. Compared with normal samples, epoxy-coated samples show sudden increase from around 100nm. The tangent of the curves is shown in Fig. 3. With and without epoxy-coating shows its peak around 45nm and 120nm, respectively, and 45nm is the threshold pore size measured with the proposed method. To confirm the obtained threshold pore size is correct, i.e., sudden intrusion of mercury occurs at around 45nm, the intrusion was stopped at different pressures, the samples were split, and the splitting surfaces were observed. The measurement was stopped at the pressures of 8.58MPa, 12.76MPa, and 43.45MPa which correspond to the pore sizes of 164nm, 100nm, and 30nm, respectively. The splitting surfaces are shown in Fig. 4. In the case of normal samples, the colour of the surface changes gradually as the pressure increases. On the other hand, when the samples are coated, sudden change occurred between 100nm and 30nm, and this corresponds to the extracted threshold pore size, 45nm. The above results indicate that with epoxy-resin-coated samples, sudden intrusion of mercury is induced at threshold pore size. Good correlation in Fig. 1 supports that the pore size at the peak in epoxy-resin-coated

164nm 100nm 30nm

(a) Normal sample



164nm 100nm 30nm

(b) Epoxy-coated sample

Figure 4. Splitting surface after mercury intrusion
(Numbers below figure indicates the applied pressure in MIP before splitting)

sample is threshold size. Normal sample also shows a peak around 120nm, however, the correlation with air or water permeability is poor, the determinant coefficients are around 0.5 or less.

WATER INFILTRATION TEST ON SPECIMENS WITH DIFFERENT THICKNESS

The time required for water penetration through concrete specimen was measured. Some specimens shown in Table 1, not all because the stock of the specimens was limited, with different thickness were used. Water sensitive sheet was attached on the bottom of the sliced cylinder specimens and water was supplied to the top side of them. The colour of the sheet changes from blue to red by water. The lateral side of the specimens were covered with aluminium tape and silicon rubber. Scotch tape was put over the water sensitive sheet. The experimental setup is shown in Fig. 5. The colour of the sheet was recorded with video cameras and time between water supply and the colour change of a part of water sensitive sheet was measured (Fig.6). Fig. 7 shows the thickness of the specimens and the required time for water penetration. Here, specimen of N70-2, W/C=70%, sealed curing until 28days after casting, was prepared during the same period in the same way with specimens shown in Table 1. All types of specimens show exponential increase of required time with the increase of the thickness and the increasing rate is much different depending on mixing design or curing condition. Since water infiltration rate in a straight cylinder tube is expressed as Eq. 1, the required time for penetration is proportional to the square of thickness and the reciprocal of the pore size.

$$x = \sqrt{2r\gamma t \cos\theta / 3\mu} \qquad (1)$$

Here, x, r, γ, t, θ, and μ are infiltration length, channel radius, boundary tension between air and liquid, time, contact angle, and viscosity coefficient, respectively. Fig.8 shows the relationship between required time and an indicator of water infiltration, the square of the specimen thickness divided by threshold pore size shown in Table 1. The plots distribute in a line, however, the determination coefficient is moderate. Threshold pore size shown in Table 1 was measured with MIP excluding coarse aggregate. In concrete specimen, however, there is transition zone around coarse aggregate and it affects mass transfer because transition zone has larger pore. As a result, the smallest pore size for penetration increases if the thickness of specimen is around or less than coarse aggregate size. In Fig. 8, such change is not considered, same threshold pore size is used regardless of the thickness of specimens, and that may be the reason of such deviation. In the next chapter, surface air permeability on

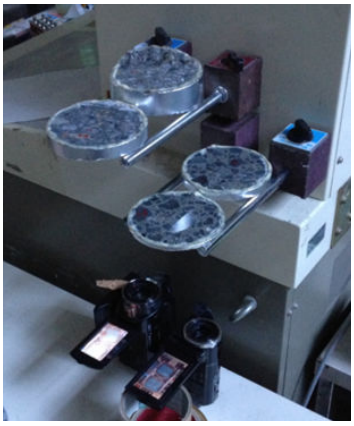


Figure 5. Experimental setup

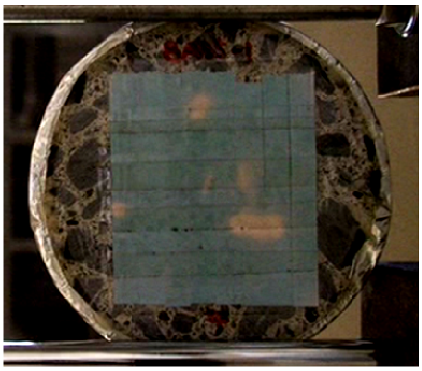


Figure 6. Colour change of water sensitive sheet

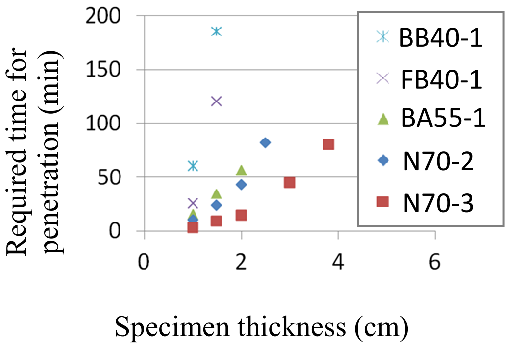


Figure 7. Specimen thickness and required time for water penetration

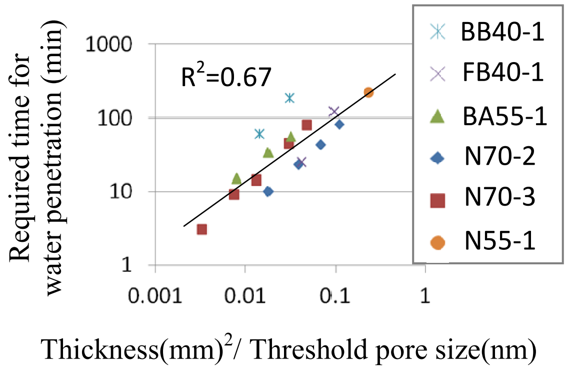
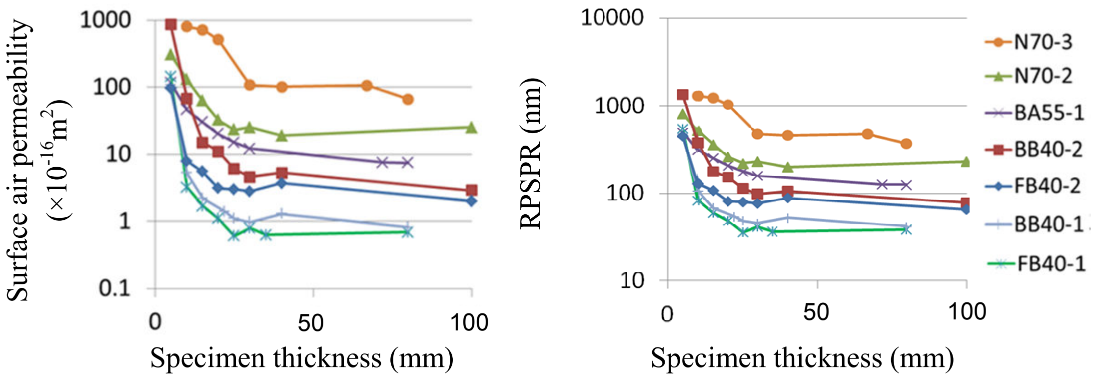


Figure 8. Required time and indicator of infiltration (Threshold pore size from Table1)

specimens with different thickness is conducted to estimate RPSPR of them.

SURFACE AIR PERMEABILITY TEST WITH DIFFERENT THICKNESS SPECIMEN

RPSPR is the smallest pore which mass must pass through to penetrate the specimen. As the thickness of the specimen becomes less, there is more possibility for mass to penetrate it avoiding smaller pore and, as a result, RPSPR becomes larger. To understand the change of RPSPR depending on the thickness of specimens, surface air permeability test was conducted on the specimens with different thickness, assuming linear relationship between air permeability and RPSPR. Here, in the case of thin specimen, the thickness is less than "effective depth", a parameter obtained in surface air permeability test. However, "effective depth" is just calculated from the surface air permeability which is derived from time and pressure change, in other word, the effective depth doesn't indicate any physical extent of the measurement. Based on the above understanding, the results of surface air permeability test were regard as valid even when the thickness of the specimen was less than "effective depth". The test results are shown in Fig. 9(a). Air permeability decreases as the thickness increases, however, when the thickness is more than 3cm, the values are converged. Air permeability is converted to RPSPR with Eq. 2 and the results are shown in Fig. 9(b).



(a) Surface air permeability (b) Calculated RPSPR

Figure 9. Surface air permeability and calculated RPSPR

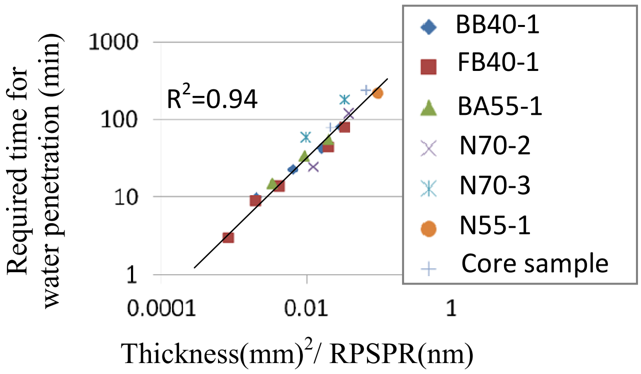


Figure 10. Relationship between required time and indicator of water infiltration (RPSPR is calculated from Eq. 2)

$$[\text{RPSPR (nm)}] = 46\times[\text{Surface air permeability } (\times10^{-16}\text{m}^2)]^{0.5} \quad (2)$$

The equation is derived by regression analysis of Fig. 1(a). In Fig. 9(b), the less specimens' thickness is, the more RPSPR increases when the thickness is less than 25mm. The tendency indicates that when the thickness of a specimen is less than a certain value, the smallest pore size which mass have to pass through to penetrate increases since there is more possibility for mass to avoid such small pores. The results in Fig. 9 show that RPSPR changes depending on the thickness of specimen if the thickness is less than a certain value, and also, the same depth of the boundary, 25mm, shows that this change is not due to "effective depth" since it must be different depending on concrete permeability. Using RPSPR shown in Fig. 9 (b), indicator was calculated, as in Fig.8, and shown in Fig. 10. In the figure, the results of two core samples (Takahashi et al., 2010) taken from a sea wall of fly ash concrete (W/C=46, 47%), thickness is 15mm and surface air permeability is 0.53m^2 and 5.4m^2, are also plotted. The correlation between the indicator and the time for water penetration is very high regardless of mixing design, curing condition, etc. The above results indicate that, first, the change of RPSPR depending on specimen's thickness is captured by surface air permeability test, and, RPSPR can be an indicator of water infiltration when the concrete is enough dry. The mechanism of this high correlation will be discussed in future in detail. Non-destructive testing methods to evaluate threshold pore size and RPSPR are now under development.

CONCLUSION

In this paper, first, it was confirmed that epoxy-resin coating on MIP sample causes sudden intrusion at threshold pore size. Secondly, it was shown that representative pore size of permeation resistance (RPSPR) can be an indicator of water infiltration rate in concrete when the concrete is enough dry. It is also shown that surface air permeability test on specimens of different thickness can estimate RPSPR properly which varies with their thickness. The above outcome indicates that when one of the following value, threshold pore size or RPSPR, water permeability, or, if the concrete is enough dry, air permeability, or infiltration rate is obtained, other values can be estimated. In the future, non-destructive testing method will be proposed to obtain above indicators.

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