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Study on some factors affecting the results in the use of MIP method in concrete research

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Abstract

Effects of rate of pressure application and forms and type of sample on porosity and pore size distribution of concrete estimated through mercury intrusion porosimetry (MIP) are presented in this experimental work. Two different forms of concrete sample, namely, crushed chunks of concrete and small core drilled out from the concrete beam specimens, were used for this study. The results exhibit that the rate of pressure application in mercury porosimetry has little effect on porosity and pore size distribution of concrete. It is also demonstrated that small cores drilled out from large concrete specimens are preferable as samples for performing porosimetry test on concrete.

Keywords: Mercury porosimetry; Porosity; Pore structure; Pore size distribution; Concrete

1. Introduction

Until recently, the knowledge of nature and behaviour of concrete was restricted to a few phenomenological properties. In the past few decades, concrete technologists have focused their attention on the internal structure of concrete. The internal structure of concrete represented by its pore system, i.e., the porosity and pore size distribution, plays a decisive role in influencing the physical and mechanical properties of concrete. The most valued properties such as compressive strength and durability of hardened concrete are directly related to its pore structure. Mercury intrusion porosimetry (MIP) is the most widely adopted method for the study of pore structure characteristics of cement-based materials. In recent years, this method has been widely used for the study of pore structure of hydrated cement paste, mortar and concrete [1–12]. However, mercury porosimetry results are affected by a number of experimental factors such as contact angle and surface tension of mercury, sample preparation, forms and types of sample, sample-drying technique and rate of pressure application, etc. [1,8,9,11,13–22]. Hence, it is necessary to select the form and types of sample, sample-drying techniques, contact angle value of mercury, etc., suitably, so that the results are obtained with minimal error. While using this technique for the study of porosity and pore size distribution of cement-based materials, approaches adopted by different research workers in this regard vary markedly from one another [1,7,8,14,16,17,19,22–27]. Effect of various sample-drying techniques on MIP results has been studied extensively in the past by a number of research workers [8,15,19,20]. The effect of sample type on the other hand has got much less attention. The contact angle between cement-based materials including concrete and mercury has been measured by many in the past and adopted accordingly depending upon the method of sample-drying technique used. Similarly, surface tension value of mercury used in Washburn equation for calculation of pore sizes has been adopted based on measured values. However, effect of rate of pressure application on MIP results has been investigated only to a limited extent [19]. Effect of pore shape and other minor factors, such as effect of mercury compression, has been also studied by some workers in the past [28].

In the past, pore structure study of concrete has been carried out using porosimetry test performed on either cement paste or mortar extracted from concrete [29,30] Mortar samples were again two types, namely, extracted chunk of mortar and drilled small mortar core, both

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obtained from the parent concrete [8,29]. Winslow and Liu [9] reported that the pore size distribution of cement paste measured by MIP depends on the method of preparation of the paste. Pore size distribution of plain paste prepared independently is different than that of a paste separated from fresh mortar of concrete, even though the pastes were identical otherwise. Cement paste present in mortar and concrete is more porous than the plain paste prepared independently. The studies carried out by Kumar [30] and Laskar et al. [31] have demonstrated that the mortar adhered with coarse aggregate is more porous than the mortar devoid of coarse aggregate taken from the same concrete specimen. This is due to the fact that the transition zone present between the boundary of coarse aggregate and the bulk mortar matrix is also included in the sample containing the coarse aggregate. This transition zone is more porous than the bulk mortar present in the interior concrete. Thus, it was concluded that mercury porosimetry test performed on concrete should be conducted using concrete sample, i.e., mortar adhered with coarse aggregate, rather than plain mortar devoid of coarse aggregate extracted from the parent concrete [30,31]. Sample-to-sample variation is likely to be more when such chunks of concrete are adopted in testing. Small cores, drilled from the concrete element on the other hand, are likely to preserve the sample-to-sample uniformity more reliably. In the present work, two forms of sample are considered, namely, chunk of concrete obtained by crushing large-sized core and small cores drilled directly from concrete beam specimen. The results of the investigation on the effects of the above two forms of sample and rate of pressure application on pore size distribution of concrete determined through MIP are presented in this paper.

2. Research program and experimental details

2.1. Starting materials

The cement used in this work was ordinary Portland cement. The aggregates used were graded 20 mm nominal maximum-sized crushed quartzite with reddish, land quarried sand confirming to zone II of British standard. The water used was potable laboratory tap water.

2.2. Test specimens

The experimental program consisted of casting concrete beams of dimensions 100 × 200 × 1000 mm. The mix proportions used are given in Table 1. These mix proportions were designed to have a 95% characteristic cube strength varying from 15 MPa for Mix 1 to 40 MPa for Mix 6 to cover the usual range of normal-strength concrete. The specimens were demolded 24 h after casting and were cured in moist environment for 27 days by wrapping the specimens all around with wet Hessian cloth. The Hessian cloth was constantly maintained wet by sprinkling water on it.

Table 1
Concrete mix proportions and mix designations

Mix designation	Proportions of			
	Cement	Fine aggregate	Coarse aggregate	Water/cement ratio
Mix 1	1	2.5	5.1	0.65
Mix 2	1	2.2	4.2	0.56
Mix 3	1	1.8	3.9	0.51
Mix 4	1	1.5	3.6	0.46
Mix 5	1	1.3	3.2	0.42
Mix 6	1	1.1	2.7	0.38

2.3. Sample preparation for MIP investigation

Concrete samples representative of the overall quality of concrete in the beam specimens for the forms of sample mentioned earlier were collected as described below.

Concrete chunk samples were obtained by crushing cores 75 mm in diameter and 100 mm in height. These concrete cores were drilled from the concrete beam specimens and were crushed using a Universal testing machine. Crushing was performed at the highest rate of loading, i.e., 22 MPa/min, the maximum possible with the machine used. It was expected that relatively higher rate of loading would minimize the chances of large-scale crack propagation before failure [32]. Thus, chance of formation of additional cracks was minimized. From the concrete so obtained after crushing the cores, chunks of concrete were chosen as samples for MIP test. Samples were selected ensuring presence of at least one piece of maximum-sized coarse aggregate in each chunk with sufficient quantity of mortar adhered at its surface. A few of such chunks were used in a sample, such that the quantity was just sufficient to fill the sample cell of the porosimeter and the mass of the sample so selected was about 18–20 g. Small core samples were obtained by direct drilling. Different locations on the beam specimens were chosen randomly on all surfaces of the beam for this purpose. The diameter of such cores were 25 mm and the length varying between 15 and 25 mm. Both types of sample were dried in an oven at a temperature of 105–110 °C for 24 h or more to a constant weight. After drying, the samples were kept in a desiccator till testing.

2.4. MIP testing

The porosity and pore size distribution study was carried out on a Quanta chrome Autoscan-33 mercury intrusion porosimeter capable of generating pressure in the range of subambient to 33,000 psi (227 MPa). The pore radius calculation was done by using Washburn equation, i.e., $r = -2\gamma\cos\theta/P$, where r is the pore entry radius in which mercury is being intruded, γ is surface tension, θ and P are contact angles of mercury with the solid and applied pressure, respectively. Values of γ and θ are suitably adopted from literature as described below.

2.4.1. Drying techniques, contact angle and surface tension

It is obvious from Washburn's equation that the accuracy of the measurement of pore radii is dependent on the accuracy of the adopted values of contact angle of mercury with the material of the sample and surface tension of mercury. Several factors, such as test material, sample-drying technique used, mercury purity, etc., can affect the contact angle value of mercury. For porosimetry study on cement-based materials, values of contact angles commonly adopted are 117° mostly for oven-dried sample [8,15,24], 130° for chemical drying using magnesium perchlorate hydrate [8,21] and 140° for all other techniques [19,20,24]. From a comparative investigation on effect of various sample-drying techniques on pore size distribution, Winslow and Diamond [8] reported that oven drying ensures complete removal of water unlike other drying techniques and recommended this technique as a standard method for sample drying in MIP. Similarly, surface tension value of mercury with material under investigation also affects MIP results. For the cement-based materials, the values of surface tension of mercury is reported to vary between 0.473 and 0.485 N/m [8,10,11,15,24]. This range is relatively small. Therefore, the choice of surface tension value within the reported range has a smaller effect on MIP results. The most commonly reported value of surface tension of mercury for oven-dried sample is 0.484 N/m. In the present study, the contact angle and surface tension values were taken as 117° and 0.484 N/m, respectively, because oven-drying procedure was adopted as a technique for sample drying [11,29].

2.4.2. Pore shape

Washburn equation is valid for pores of circular cross section, which is the most commonly adopted shape of the pores. Jenkin and Rao [28] have suggested a more general expression for the pores of other shapes such as elliptical or slit by introducing a shape factor in the Washburn equation, i.e.,

$$d = -\phi\gamma\cos\theta/p$$

where ϕ and d stand for the shape factor and narrow dimension of the pore, respectively, and the other symbols have the same meaning as mentioned earlier. The shape factors for various types of pore range from 2 for slit (parallel walls) to 4 for cylindrical pore of circular cross section [11]. Based on extensive work, Cook and Hover [11] reported that the shape factor has no effect on total porosity but exhibits minor influence on the threshold diameter. Further, based on scanning electron micrographs (SEM) observation, Maage [33] concluded that no other simple cross section other than circular cross section fits better to the actual cross section of the pores present in the materials. Based on this fact, the pore shape is assumed to be circular in cross section for this study. Hence, the final form of the Washburn equation used in this study is as given below:

$$r = -637,500/p$$

where p is applied pressure in psi and r is the pore radius in Å. Thus, the range of pore radii covered for subambient to 33,000 psi applied pressure was 0.002–200 μm.

2.4.3. Effect of solid compressibility, mercury compression and other minor factors

Mercury and solid sample both are compressible at very high pressures. Compression of pressure cavity and sample cell may also affect the porosimetry results at very high pressures [34–36]. Errors due these factors can be significant while dealing with soft plastics [35,36]. From the investigation conducted, it is reported that, for cement-based materials, these factors affect the pore size distribution curve only in the region below the pore diameter of 50 nm. It is further reported that error due to these factors is never more than 3% [36]. Thus, for the study of pore size distribution of concrete, effect of these factors can be neglected without introducing significant error.

2.5. Number of samples

The possible largest dimension of the sample cell restricts the size of the sample used in MIP. The maximum volume of mercury intrusion also restricts the sample size. The sample cell used in this work was cylindrical with diameter of 26 mm and height of 38 mm. Usual dimension of concrete structural element is very large compared to the above size. Therefore, due to inherent point-to-point material variability in concrete, the MIP results obtained from one sample to another are likely to exhibit wide variation. Thus, the number of samples required to be tested in order to get MIP results within a reasonable accuracy for concrete may be more than three. In an earlier paper, a thorough investigation on this aspect of evaluation of concrete through MIP was presented [31]. Three most important parameters of MIP, namely, porosity, mean distribution radius and retention factor, were considered in that investigation. These factors are closely related to the strength and performance properties of concrete. The statistical distribution of these parameters were investigated upon and ascertained to be normal. Further, it had been observed that the coefficient of variation of porosity is higher than those of mean distribution radius and retention factor and ranged between 10% and 18%. The number of samples required to obtain all the results within 15% of sampling error with a confidence level of 95% were estimated to be 6 based on Stein's two stage formula [31]. However, when three samples are used, the sampling error would be within ±25% with a confidence level of 95%. Many earlier researchers reportedly used 2 or 3 number of samples for MIP test [9,10]. To study the effects of number of sample on porosity and pore structure, six samples were thus used to obtain average porosity, mean distribution radius, etc. However, in the study of effect of the rate of pressure application, three samples were used expecting a maximum error of ±25%.

3. Effect of rate of pressure application on porosity and pore size distribution

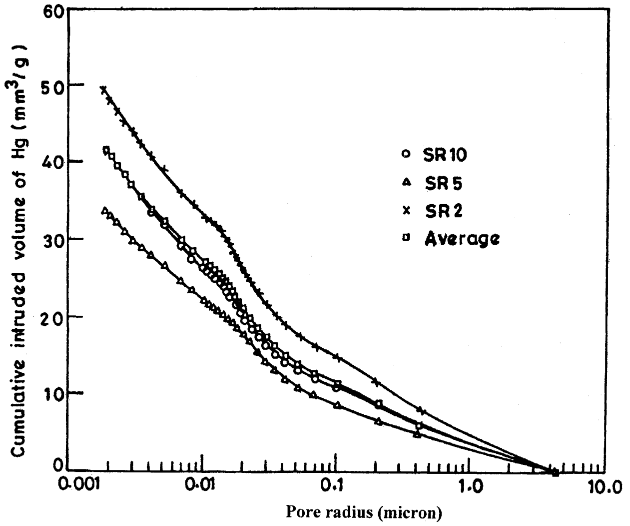

Fig. 2. Pore size distribution curves at different scanning rates of machine for crushed samples taken from concrete of Mix 1.

Most of the commercially available porosimeters have the facility for applying pressure at varying scanning rate, while some porosimeters can also apply pressure by equilibrating at each pressure step. To investigate the effect of rate of pressure application on porosity and pore size distribution of concrete, porosimetry tests were carried out on the samples of concrete at different scanning rate of machine, i.e., the rate corresponding to the machine knob position of 2, 5 and 10. In the machine, the range of scanning rate is divided on a scale of 0–10 corresponding to 10 positions of the knob. Scanning rate of machine controls the rate of pressure applied to intrude mercury into the pores of sample. Thus, by considering scanning rates corresponding to positions 2, 5 and 10 of the machine knob, the range of scanning rate available was nearly covered. Further, to detect any possible interaction effect of concrete mix characteristics and rate of pressure application on porosimetry results, this investigation was carried out on concrete of two different mixes, namely, Mix 1 and Mix 6 corresponding to the water/cement ratios of 0.65 and 0.38, respectively. Mix 1 and Mix 6 represent the lowest strength and the highest strength of concrete used in this study. Similarly, to study any possible interaction between the rate of pressure application and the different forms of sample, namely, crushed and cored sample of concrete, the tests were also conducted on both forms of samples extracted from the same concrete beam specimens. Thus, four varieties of samples were used in this investigation and at each scanning rate, i.e., at each rate of pressure application. Triplicate samples of each of the concrete specimens were tested at each of the rate of pressure application. The average pore size distribution curves for these samples were obtained by averaging the cumulative intrusions of all the three samples at a given radius of the pore and plotting the resulting average cumulative intrusion against pore radii. The resulting average pore size distribution curve was considered as the representative pore size distribution for the concrete under investigation. This representative pore size distribution curve was used for the calculation of

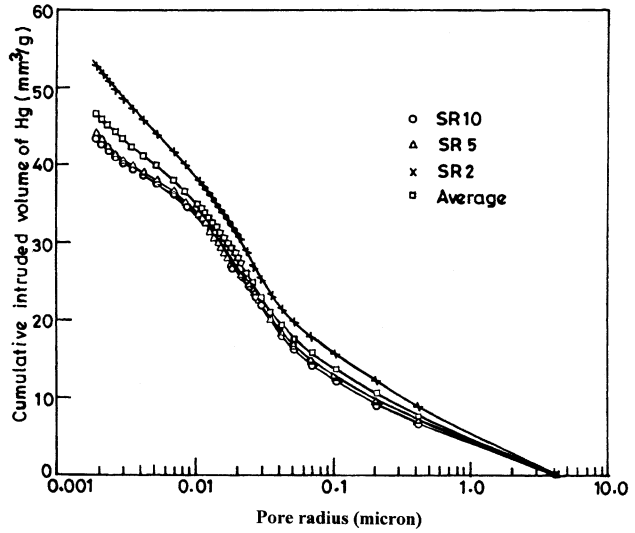

Fig. 1. Pore size distribution curves at different scanning rates of machine for cored samples taken from concrete of Mix 1.

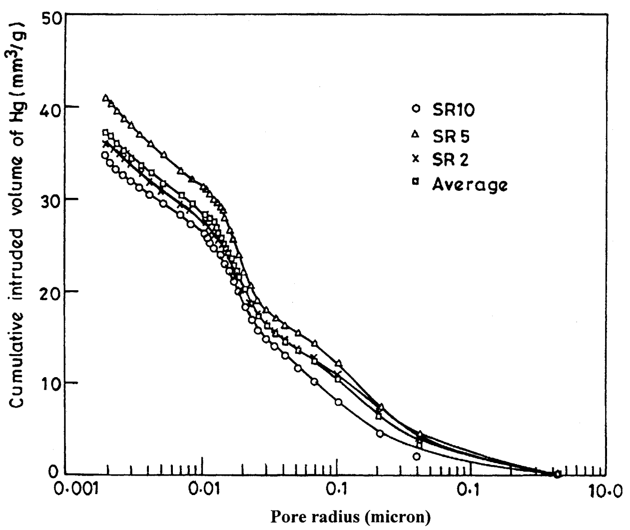

Fig. 3. Pore size distribution curves at different scanning rates of machine for cored samples taken from concrete of Mix 6.

relevant pore structural parameters such as porosity, mean distribution radius and equivalent pore radius, etc. Figs. 1–4 present the average pore size distribution curve at each of the selected scanning rate. Fig. 1 shows the average pore size distribution curve of the concrete for the cored samples extracted from the beam specimen of concrete cast with Mix 1. A similar figure for crushed sample extracted from the same beam is given in Fig. 2. Figs. 3 and 4 correspond to the cored and crushed samples, respectively, taken from the beam cast with concrete designated as Mix 6. A close observation of Fig. 1 reveals that the maximum cumulative intruded volume of mercury per gram of the sample and cumulative intruded volume of mercury at any radius for the scanning rate of 2 is more than that of scanning rate of 5, which is again more than that corresponding to scanning rate of 10. Similarly, in Fig. 2, it is obvious that the maximum cumulative intrusion corresponds to scanning rate of 2, but unlike Fig. 1 the minimum cumulative intrusion volume per gram corresponds to the scanning rate of 5. Further, in Fig. 3, the maximum cumulative intrusion per gram of sample is highest for scanning rate of 5 and lowest for scanning rate of 10, whereas in Fig. 4 the same maximum corresponds to scanning rate of 2 and the minimum corresponds to scanning rate of 10. However, the nature of these pore size distribution curves at different scanning rates is similar. On comparison of these pore size distribution curves, it is apparent that the rate of pressure application affects the pore size distribution of concrete somewhat randomly. The pore size distribution curves representing the average intrusion of three scanning rates are also shown in Figs. 1–4. The observed maximum deviation of the cumulative intrusion from their average at any radius due to scanning rate is ± 17%, which is reasonably small. Therefore, it is possible to select a single scanning rate at which comparative study of porosity and pore size distribution of concrete can be performed through porosimetry, as rate of pressure application has a little effect on porosimetry results. Hearn and Hooton [19] also arrived at a similar conclusion earlier from their investigation. Slower rates are highly time consuming. Therefore, in the rest of this work, i.e., to know the effect of the forms and types of sample of concrete on its porosity and pore size distribution, a moderate rate of pressure application, i.e., corresponding to machine knob position 5, was adopted. It is worthwhile to mention here that the time required for one

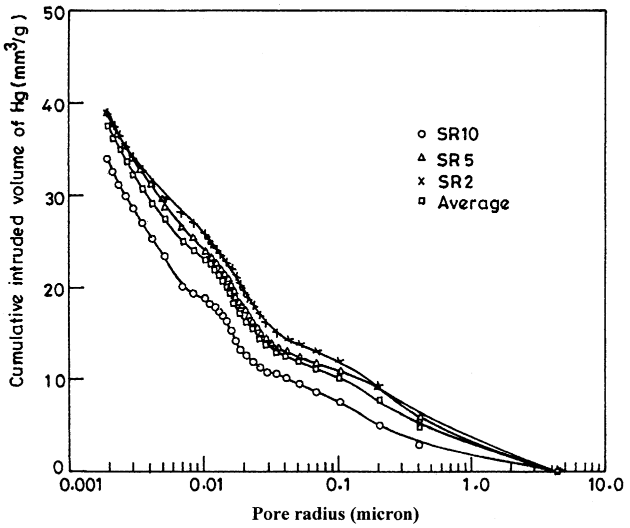

Fig. 4. Pore size distribution curves at different scanning rates of machine for crushed samples taken from concrete of Mix 6.

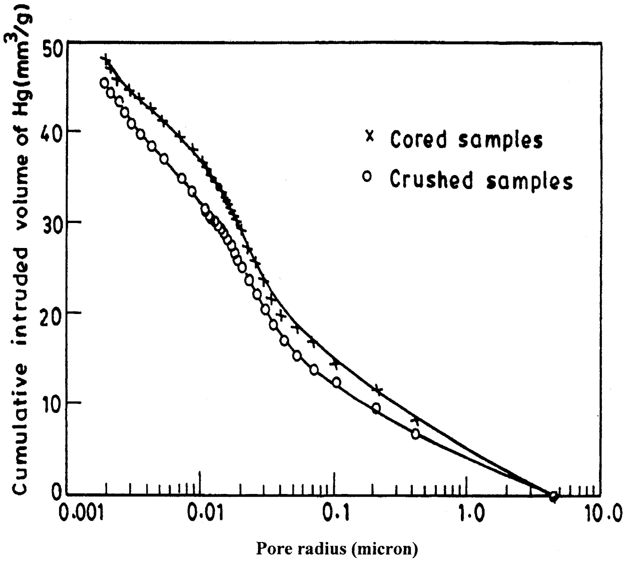

Fig. 5. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 1.

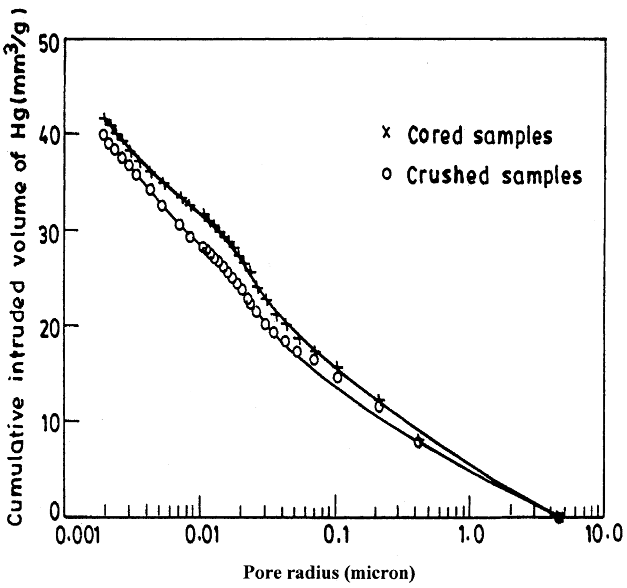

Fig. 6. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 2.

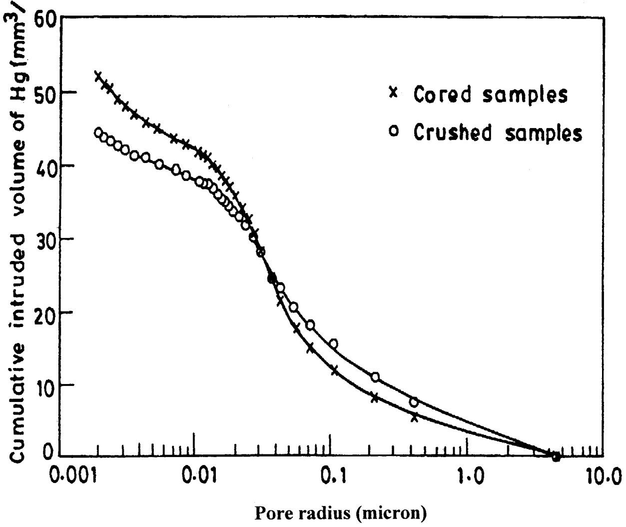

Fig. 7. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 3.

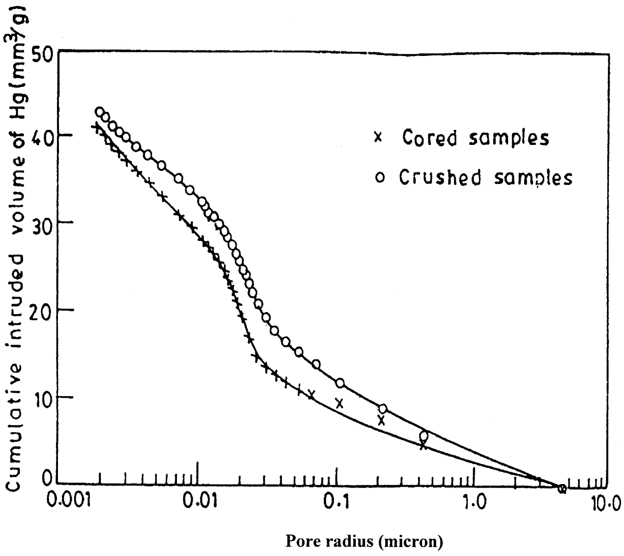

Fig. 9. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 5.

cycle of intrusion and extrusion curve at scanning rates of 2 and 5 are 80 and 40 min, respectively.

4. Effects of forms of sample of concrete on its porosity and pore size distribution

To investigate on the effect of forms of sample on porosimetry results, two types of sample, i.e., directly drilled cored sample and crushed chunk sample of concrete, were adopted. These samples were prepared from six beams cast with Mix 1 to Mix 6, one from each of the concrete mixtures. The average pore size distribution curves for crushed samples and cored samples of concrete are shown in Figs. 5–10. From the figures, it is obvious that both forms of concrete sample yield approximately the same value of total cumulative intrusion of mercury. More precisely, the difference in porosity value is seldom more than 8%, except in case of concrete of Mix 3 where the same is of the order 14%. Thus, the concrete sample whether cored or crushed provides nearly identical MIP results. Therefore, it is obvious from the study that either form of the concrete sample can be used in MIP study. However, in order to obtain crushed concrete samples, the larger-sized core is required to be drilled from the actual structure, which when crushed under short-term loading results in the desired samples. On the other hand, extraction of small core sample from structure is a one-step procedure. Further, in cored

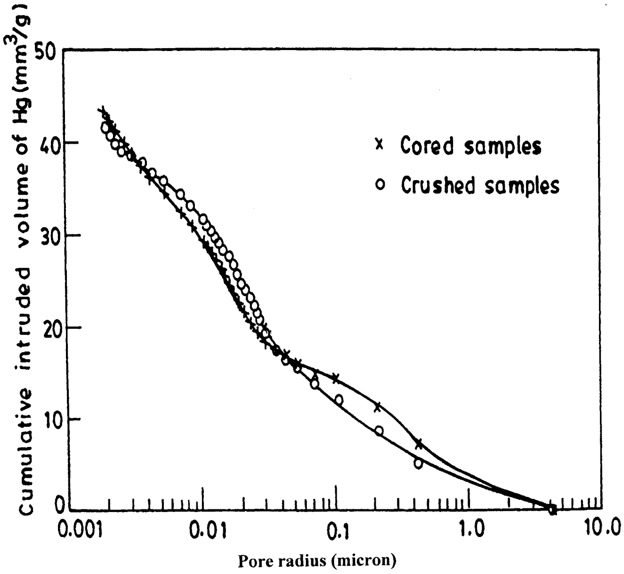

Fig. 8. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 4.

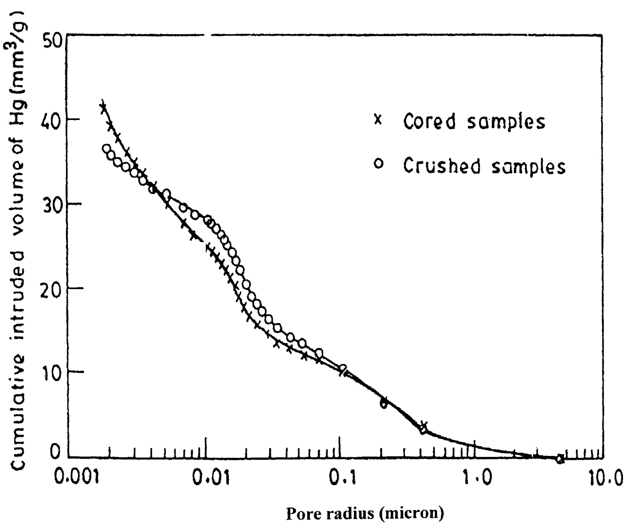

Fig. 10. Pore size distribution curves for cored and crushed samples taken from concrete of Mix 6.

Table 2
Average porosity and pore structural parameters of concrete based on cored and crushed samples

Mix no.	Cored sample					Crushed sample				
	Porosity (p)		Mean distribution radius (r_m) (μm)	Mean equivalent radius (r_e) (μm)	Retention (R) (%)	Porosity (p)		Mean distribution radius (r_m) (μm)	Mean equivalent radius (r_e) (μm)	Retention (R) (%)
	Mean (%)	S.D.				Mean (%)	S.D.			
Mix 1	12.96	1.53	0.186	0.034	43.5	12.70	3.82	0.173	0.027	40.6
Mix 2	11.93	1.32	0.198	0.039	52.9	11.38	0.90	0.203	0.034	45.5
Mix 3	11.80	2.20	0.189	0.046	46.0	13.93	1.27	0.151	0.030	39.4
Mix 4	11.22	1.06	0.167	0.031	48.8	11.47	1.48	0.186	0.028	48.5
Mix 5	11.50	0.42	0.171	0.030	49.2	11.11	2.06	0.155	0.023	43.5
Mix 6	9.26	1.03	0.143	0.028	63.8	10.53	2.20	0.146	0.019	52.2

samples, similarity can be maintained in sample mass and sample dimensions, hence minimizing the sample-to-sample variation in concrete. Moreover, in the case of cored sample, it is also possible to extract the test samples from different depth in the structural concrete, while the same is not possible for crushed concrete sample. Thus, when cored samples are used, the profile of porosity and pore structure across a section of concrete can be obtained. Further, the other important pore structural parameters calculated from pore size distribution curves, namely, average porosity and its standard deviation, mean distribution radius, mean equivalent radius and average retention factors for all the six mixes obtained on both forms of concrete sample, are given in Table 2. The mean distribution radius (r_m) [31] and equivalent pore radius (r_e) [10] are defined by following equations, respectively:

$$\ln r_{\mathrm{m}} = \frac{\sum_{i=1}^{i=n} V_i \ln r_i}{\sum_{i=1}^{i=n} V_i} \tag{1}$$

and

$$r_{\mathrm{e}}^2 = \frac{\int \frac{\mathrm{d}v}{\mathrm{d}(\log r)} r^2 \mathrm{d}(\log r)}{\int \frac{\mathrm{d}v}{\mathrm{d}(\log r)} \mathrm{d}(\log r)} \tag{2}$$

where V_i is the intrusion of mercury corresponding to ith radius range represented by mean radius r_i and $\mathrm{d}V/\mathrm{d}(\log r)$ is the differential pore size distribution with respect to log r, r being the radius of the pore. Retention factor R is the ratio of retained volume of mercury after the first cycle of intrusion–extrusion to the total intrusion of mercury expressed in percentage. The results presented in Table 2 reveal that the standard deviations of porosity obtained from cored samples are relatively lower, and average standard deviations for all the six concrete mixtures are 1.26 and 1.96, respectively, for cored and crushed sample. Thus, sample-to-sample variation is relatively lower in case of cored sample. Hence, cored sample of concrete is the most suitable form of sample of the concrete for the study of its porosity and pore size distribution.

5. Conclusions

1. The rate of pressure application has little or insignificant effect on porosity and pore size distribution of concrete as determined through porosimetry.
2. Both crushed and cored sample of concrete can be used for the study of its porosity and pore size distribution. However, cored samples exhibit less sample-to-sample variation. Hence, from the point of view of similarity in sample mass, sample dimensions, nature of concrete contained in sample and ease of obtaining it, the cored samples of concrete is preferable.

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References

[1] P. Parcevaux, Pore size distribution of Portland cement slurries at very early stages of hydration (influence of curing, temperature and pressure), Cem. Concr. Res. 14 (3) (1984) 419–430.
[2] M. Rößler, I. OdLer, Investigations on the relationship between porosity, structure and strength of hydrated Portland cement pastes: I. Effect of porosity, Cem. Concr. Res. 15 (2) (1985) 320–330.
[3] N.McN. Alford, A. Rahman, An assessment of porosity and pore sizes in hardened cement pastes, J. Mater. Sci. 16 (1981) 3105–3114.
[4] J.J. Beaudoin, Porosity measurement of some hydrated cementitious systems by high pressure mercury intrusion—microstructural limitations, Cem. Concr. Res. 19 (6) (1979) 771–781.
[5] R.F. Feldman, J.J. Beaudoin, Microstructure and strength of hydrated cement, Cem. Concr. Res. 16 (3) (1976) 389–400.
[6] S. Diamond, A critical comparison of mercury intrusion porosimetry and capillary condensation pore size distribution of Portland cement pastes, Cem. Concr. Res. 11 (5) (1971) 531–545.
[7] A. Bentur, The pore structure of hydrated cementitious compounds of different chemical composition, J. Am. Ceram. Soc. 63 (1980) 381–386.
[8] D.N. Winslow, S. Diamond, A mercury porosimetry study of the evaluation of porosity in Portland cement, J. Mater. 5 (3) (1970) 564–585.
[9] D. Winslow, D. Liu, Pore structure of paste in concrete, Cem. Concr. Res. 20 (2) (1990) 227–235.
[10] H.W. Reinhardt, K. Gaber, From pore size distribution to an equiv-

alent pore size distribution of cement mortar, Mater. Struct. 23 (133) (1990) 3–15.

[11] R.A. Cook, K.C. Hover, Mercury porosimetry of cement-based materials and associated correction factors, ACI Mater. J. 90 (2) (1993) 152–161.

[12] F. Indelicato, On the correlation between porosity and strength in high-alumna cement mortars, Mater. Struct. 23 (135) (1993) 289–295.

[13] E.J. Sellevold, Mercury porosimetry of hardened cement paste cured or stored at 97 °C, Cem. Concr. Res. 4 (3) (1974) 399–404.

[14] R.F. Feldman, J.J. Beaudoin, Pretreatment of hardened hydrated cement pastes for mercury intrusion measurements, Cem. Concr. Res. 21 (2) (1991) 297–308.

[15] D.H. Bager, E.J. Sellevold, Mercury porosimetry of hardened cement paste, the influence of particle size, Cem. Concr. Res. 5 (2) (1975) 171–177.

[16] M. Sadegzadch, C.L. Page, R.S. Kettle, Surface microstructure and abrasion of concrete, Cem. Concr. Res. 17 (4) (1987) 581–590.

[17] B. Bourdtte, E. Ringot, J.P. Ollivier, Modelling of the transition zone porosity, Cem. Concr. Res. 25 (4) (1995) 741–751.

[18] C.G. Litvan, Variability of nitrogen surface area on HCP, Cem. Concr. Res. 6 (1) (1976) 139–144.

[19] N. Hearn, R.D. Hooton, Sample mass and dimension effects on mercury intrusion porosimetry results, Cem. Concr. Res. 22 (5) (1992) 970–980.

[20] M. Moukwa, P.C. Aïtcin, The effect of dying on cement pastes pore structure as determined by mercury porosimetry, Cem. Concr. Res. 18 (5) (1988) 745–752.

[21] A. Auskern, W. Horn, Capillary porosity in hardened cement paste, J. Test. Eval. 1 (1) (1973) 74–79.

[22] R.A. Cook, K.C. Hover, Experiments on contact angle between mercury and hardened cement paste, Cem. Concr. Res. 21 (6) (1991) 1165–1175.

[23] F.S. Rostasy, R. Weib, G. Wiedemann, Changes of pore structure of cement mortars due to temperature, Cem. Concr. Res. 10 (2) (1980) 157–164.

[24] U. Schneider, U. Diederichs, Detection of crack by mercury penetration measurement, in: F.H. Wittmann (Ed.), Fracture Mechanics of Concrete, Elsevier, Amsterdam, 1983, pp. 207–222.

[25] F. Metz, D. Knöfel, Systematic mercury porosimetry investigations on sandstones, Mater. Struct. 25 (147) (1992) 27–136.

[26] C. Ehm, U. Schneider, The fracture process of concrete at high temperature and compressive stress, in: F.H. Wittmann (Ed.), Fracture Toughness and Fracture Energy of Concrete, Elsevier, Amsterdam, 1986, pp. 41–50.

[27] R.L. Day, Reaction between methanol and Portland cement paste, Cem. Concr. Res. 11 (3) (1981) 341–349.

[28] R.G. Jenkins, M.B. Rao, Effect of elliptical pores on mercury porosimetry results, Powder Technol. 38 (1984) 177–180.

[29] A.K. Suryavanshi, R.N. Swamy, Influence of penetrating chlorides on the pore structure of structural concrete, Cem. Concr. Aggregates 20 (2) (1998) 169–179.

[30] R. Kumar, Strength and permeation quality of concrete through mercury intrusion porosimetry, PhD thesis, Department of Civil Engineering, Indian Institute of Technology Delhi, 1997.

[31] A.I. Laskar, R. Kumar, B. Bhattacharjee, Some aspects of evaluation of concrete through mercury intrusion porosimetry, Cem. Concr. Res. 27 (1) (1997) 93–105.

[32] P.K. Mehta, Concrete: Structure, Properties and Materials, Prentice-Hall, New Jersey, 1995.

[33] M. Maage, Frost resistance and pore size distribution in bricks, Mater. Struct. 17 (101) (1984) 345–350.

[34] D. Manhoman, P.K. Mehta, Influence of pozzolanic slag and chemical admixtures on pore size distribution and permeability of hardened cement pastes, Cem. Concr. Aggregates 3 (1) (1981) 63–67.

[35] S. Lowell, J.E. Sheilds, Powder Surface Area and Porosity, Chapman & Hall, London, 1984.

[36] T. Allen, Particle Size Measurement, Chapman & Hall, New York, 1981.