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## SOME ASPECTS OF EVALUATION OF CONCRETE THROUGH MERCURY INTRUSION POROSIMETRY

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### ABSTRACT

Through an experimental investigation, statistical distribution of three identified parameters of mercury intrusion porosimetry which are closely related to the strength and performance of concrete, have been worked out. It has been demonstrated that distribution of these three parameters are normal for different mix proportions of concrete. The minimum number of samples to be tested while evaluating performance of concrete through these parameters of mercury intrusion porosimetry have been estimated using the experimental data for various permissible errors at 95% confidence level. Further, the number of samples to be tested for permissible errors to be within 15% of the true value are also suggested. In the second part of the paper an investigation on the form of sample to be adopted for MIP study of in-situ concrete has been presented, whereby it has been demonstrated that it is preferable to use mortar adhered with aggregate, rather than mortar devoid of aggregate from the same parent in-situ concrete.

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### Introduction

Mercury intrusion porosimetry (MIP) is a popular method for studying porosity and pore structure of cement based materials. The method has been successfully used for many studies on hydrated cement pastes (hcps) and cement mortars (1-6). In recent years the applicability of the method has been extended to concrete as well (7,8). However, while using MIP study on concrete, one must keep in mind that the MIP results are affected by the method of sampling, sample conditioning, sample mass and dimension used, the rate of pressure application, assumed pore shape, and values of contact angle and surface tension of mercury assumed, etc. The MIP results may also be affected by the expansion of sample cell under pressure, differential mercury compression, sample compression and hydrostatic head of mercury. Some of the above effects have been recognized in past and relevant correction factors have been sug-

gested by Cook and Hover (8). Hearn and Hooton (9) reported the effects of sample mass, sample dimensions and rate of pressure application on pore size distribution results obtained through MIP study conducted on cement paste samples. The effect of different techniques of sample conditioning on pore size distribution of cement mortar has been reported by Konecny and Naqvi (10). However, while extending the use of MIP for pore structure studies on concrete, two other factors assume significant importance. One such factor is the variability of concrete material from one sample to another and the consequent variation in MIP results. The size of the sample used in MIP is restricted by the possible largest dimensions of the sample cell used. The conventional size of the largest cylindrical sample cell is 25 mm in diameter with 35 mm in height, by which the maximum size of the sample to be used in MIP is limited. Dimensions of concrete structure are very large compared to the above maximum possible sample dimensions, and due to inherent point to point material variability in concrete, the MIP results obtained from one sample to another is likely to be widely varying, even though the samples might have been collected from near by locations in a structure. Other conventional tests on concrete are performed on larger samples, thus in order to get an accuracy similar to those of conventional tests, the number of the samples to be tested for pore structure studies on concrete through MIP, would be significantly more, and thus the decision regarding the exact number of samples to be tested while adopting MIP studies for concrete assumes paramount importance.

Another, factor that assumes importance while adopting MIP for pore structure study of concrete is related to the form of the samples, to be used in the porosimetry experiment. The sample conventionally used for MIP study can be either powdered (3,9), granular chunks (3,4,9) or specimen cast to the same dimension and shape as that of the sample cell itself (6). In order to know pore structure of concrete however, one may use, the mortar chunks extracted from the parent concrete (3,4,9) or a number of concrete chunks inclusive of the aggregates. The cumulative intrusion volume result per unit mass is likely to be different depending upon the form of the sample. The porosity obtained using mortar chunk alone is likely to be higher, as aggregates are relatively less porous, besides, the cumulative intrusion volume obtained is likely to be devoid of interfacial shrinkage cracks in concrete. In this paper results of an experimental investigation are presented whereby, the minimum number of concrete samples required to be tested, in order to adopt MIP for evaluation of pore structure of concrete has been estimated using statistical method. Further, the necessity of using concrete chunks inclusive of coarse aggregate, instead of extracted mortar, in MIP study for evaluation of pore structure of concrete is also demonstrated.

### Parameters of MIP

From MIP study one obtains the intrusion-extrusion curves within a specific pressure range depending upon the equipment capability and hence within a specific radii range as governed by Washburns equation. From such curves, one can calculate a number of parameters, such as critical pore radius (11), mean distribution radius (12), equivalent pore radius (13), porosity, pore size distribution, surface area distribution, pore population etc., (14). The choice of such parameters would depend upon the basic purpose for which MIP study is being undertaken. According to Cook and Hover (8), the pore structure of concrete affects its behaviour in terms of strength, durability and permeability. Thus given the information about the pore structure, it is possible to evaluate the performance of concrete with certain degree of accuracy. Several models have been suggested in past which relate some of the parameters of MIP with strength

and permeability of porous material in general and hcp, mortar and concrete in particular (5,6,12-13,15-21). In general these parameters of MIP deal mainly with three aspects of pore structure, namely; the relative volume of pores that is the degree of porousness of the concrete, the average pore size and the nature of the pore, characterized by the relative volume of nearly enclosed pores. The parameters of MIP which quantify the above aspects of pore structure are: i) Total porosity ( $p$ ), ii) Mean Distribution Radius ( $r_m$ ) and iii) Retention Factor ( $R$ ). These three parameters therefore can provide the necessary information regarding the likely performance of concrete and are defined below:

The total porosity is defined conventionally as the ratio of the total volume of pores to the total volume of solid given by,

$$P = \frac{V_p}{V} \quad (1)$$

Where,  $V_p$ , is the volume of pores and  $V$  is the total volume of pores and solid. The value of 'p' can be estimated from the cumulative intrusion volume and relevant weight measurements. The mean distribution radius (12) is defined through the following equation as:

$$\ln r_m = \frac{\sum_{i=1}^n V_i \ln r_i}{\sum_{i=1}^n V_i} \quad (2)$$

Where, for the continuous intrusion curve divided in to 'n' discrete radii ranges,  $V_i$ , is the incremental intrusion of mercury corresponding to 'i' th radius range represented by the mean radius  $r_i$ . Retention factor  $R$  is the ratio of retained volume of mercury after the first cycle of intrusion-extrusion to the total intrusion volume of mercury.

These parameters of MIP therefore must be estimated with sufficient accuracy in order to adopt MIP for pore structure study of concrete. The number of samples required to be tested in order to ensure required accuracy can be ascertained statistically and this analysis is presented in the next section.

### Number of Test Samples

The procedure of ascertaining the minimum number of samples required to be tested, to ensure a reasonably accurate test result, is based on following statistical principles. Two cases may be encountered, namely; (i) when the mean, ' $\mu$ ' and the standard deviation, ' $\sigma$ ' of the population is known and, (ii) when the population mean and standard deviation are unknown and inferences are to be drawn from small number of samples. In the first case the number of samples required can be computed when the required accuracy that is the length of the confidence interval is known and can be computed from the equation-3 given below(22).

$$n = \frac{(z_{\alpha})^2 \times \sigma^2}{d^2} \quad (3)$$

Where,  $z_{\alpha/2}$ , is the standard normal variate for the given confidence interval expressed as  $(1-\alpha)$ ,  $d$  is defined later.

For the second case, Stein's formula (23,24), given in equation-4 may be used, where it is assumed that the number of the sample is relatively small and the parent population is normally distributed;

$$n = \frac{t_1^2 \times s^2}{d^2}$$

Where,  $n$  is the required number of samples,  $t_1$  is the tabulated student's  $t$ -value for the desired confidence level and follows  $t$  distribution for  $n-1$  degrees of freedom,  $d$  is the half width of the desired confidence interval, and  $s$  is the standard deviation of the sample. The ' $d$ ' can be further expressed as:  $d = E \times \mu$ , where,  $E$  is the permissible error ( $\pm\%$ ), and  $\mu$ , is the true value, that is the mean of the population.

In order to verify the assumption that the above parameters of MIP are normally distributed an experimental programme was undertaken together with statistical analysis for goodness of fit and is discussed as follows.

### Experimental Programme

**Mix Proportions and Materials.** Three mixes of concrete using commonly used materials such as; ordinary portland cement, sand conforming to zone II and 20 mm maximum size of coarse aggregate (m.s.a.) were designed to represent three different strengths. The water used was ordinary tap water. For each mix proportion, 6 standard 15 cm cubes were cast and cured in clear water tank. The 28 days cube strength was determined in standard manner using three of the cubes and the nomenclature adopted for different concrete mixes according to their strengths are shown in Table 1.

**Sample Preparation and Conditioning.** For all the three concretes designated as concrete 1, 2 and 3, the remaining three cubes were used for obtaining samples for MIP tests. The method of sampling that was adopted consistently for all the samples is as follows. The

TABLE 1  
Nomenclature Adopted

Avg. comp. strength at 28 days (N/mm <sup>2</sup> )	Mix Designation
14.00	Concrete 1
18.80	Concrete 2
33.10	Concrete 3

cubes were crushed at a relatively higher rate of load application (approximately 22 N/mm<sup>2</sup>/min), compared to that used in normal cube crushing (12-14 N/mm<sup>2</sup>/min), so that under short term loading the chance of micro crack propagation is minimum (20), and the broken chunks obtained after crushing the cubes were selected as the samples for MIP. Further, as it is well known that the interface between mortar and aggregate is different than either of the components of concrete (20), the above broken chunks were selected ensuring the presence of at least one mortar aggregate interface in each. The sample was loosely packed with a number of such chunks and the mass of sample so packed was between 10 to 18 gms. The chunks were dried before testing in an oven for 24 hours at 105°C - 110°C and there after stored in a desiccator for the spreaded period over which the testing was performed and the contact angle was assumed accordingly (7,8). Cylindrical pore shape has been assumed uniformly throughout this investigation. The differential mercury compression and sample compression were assumed to have similar affect on all the samples and are unlikely to have significant impact on the results of this investigation, therefore were ignored.

**Testing.** Testing was performed with Quantachrome Autoscan-33 mercury porosimeter having a pressure range from sub-ambient to 33000 psi. Assuming the contact angle and

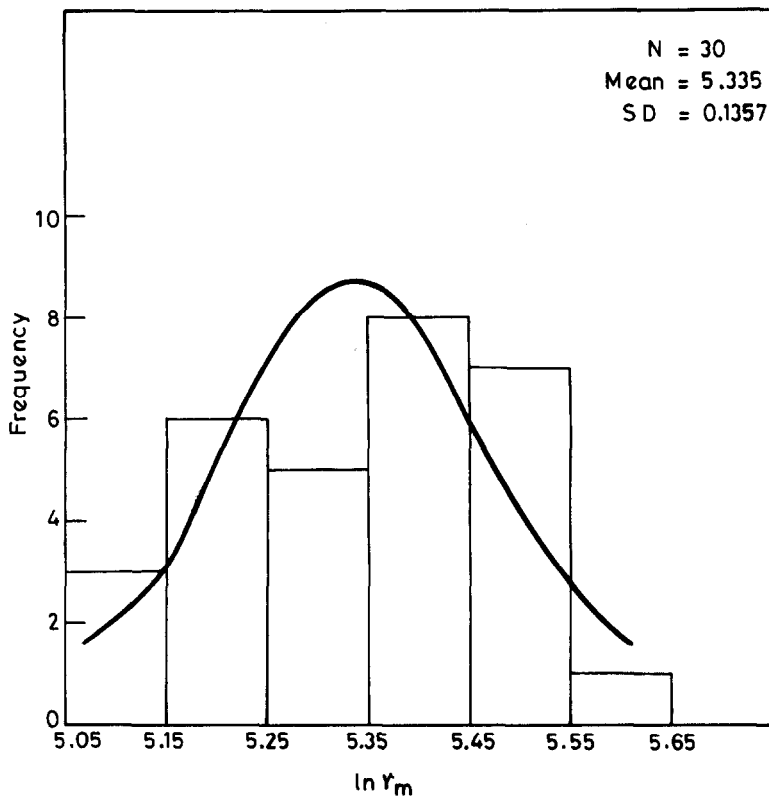


FIG. 1.  
Histogram of log - mean distribution radius (concrete 2).

the surface tension of mercury as  $117^{\circ}$  and  $0.484 \text{ N/m}$  respectively, for the oven dried samples (7,8), the expression for the pore radius according to Washburns equation, is as follows:

$$r = -637500/p \quad (5)$$

where,  $p$  is in psi and  $r$  is in Angstrom.

The sample cell fitted with the base cell of capacity  $17.7 \text{ cc.}$  was used throughout the experimental programme and all the tests were performed at a constant medium scanning rate indicated by point 5 of the machine knob on its 0-10 scale. The values of  $\ln(r_m)$ , porosity and  $R$ , mentioned earlier were calculated from the first intrusion curve so obtained. In total 30 samples were tested from each of the concrete mix 1, 2 and 3 ensuring equal number of samples from all the 3 cubes, so as to obtain a stable data for establishing statistical distribution.

**Analysis.** From 30 intrusion curves obtained for a given grade of concrete, values of all the three parameters were calculated and the raw data thus obtained for each parameter was grouped into 6 class intervals according to the formula  $i = 1 + 3.3 \log_{10} N$  (20) and the histograms were plotted, where  $N$ , stands for the number of samples and is equal to 30. The typical histograms are shown in Figs. 1-3. To confirm the normality, these distributions

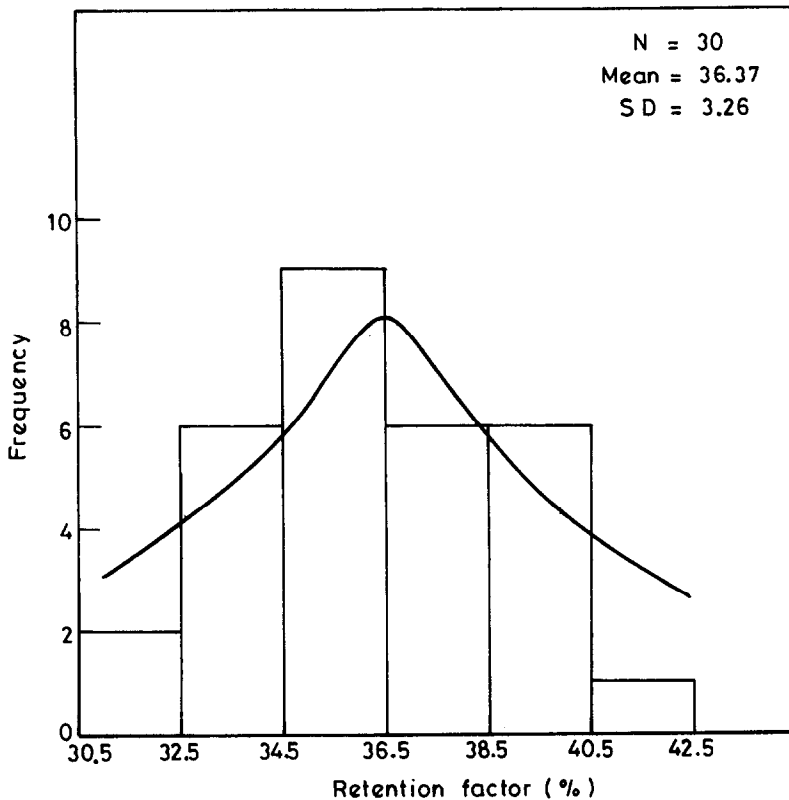


FIG. 2.  
Histogram of retention factor (concrete 2).

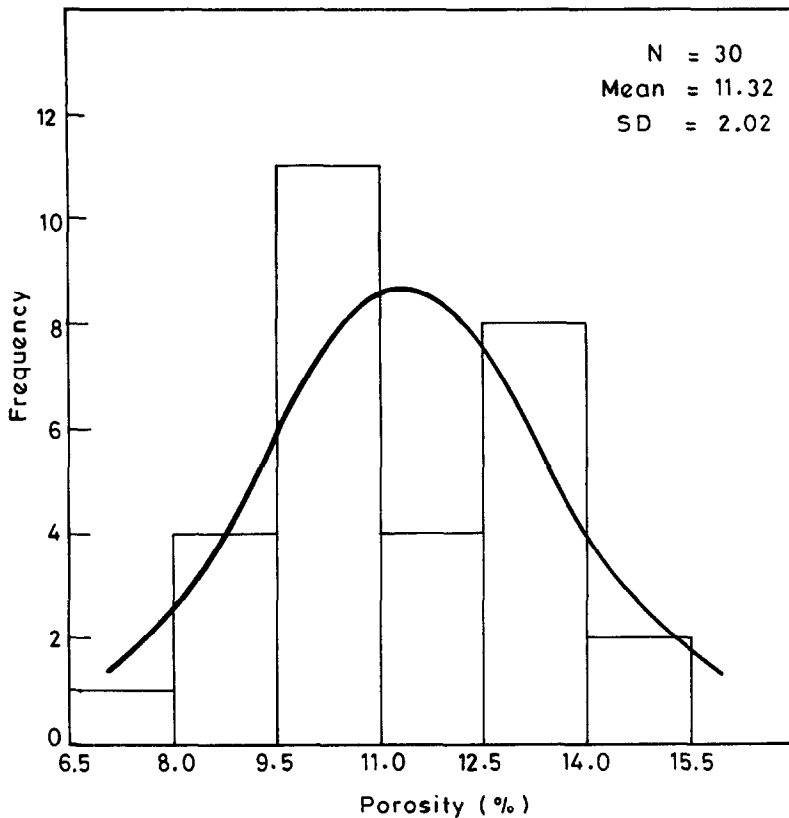


FIG. 3.  
Histogram of total porosity (concrete 2).

were tested for 'Goodness of fit' by Chi-square test (22), at 5% significance level. The results of this statistical analysis are presented in Table 2, which confirm the normality of the population. The resultant fitted normal distributions are also shown in the Figures 1-3.

Having assured the normality of the population for  $p$ ,  $\ln(r_m)$  and  $R$ ; the number of samples required to be tested in order to ensure a desired degree of accuracy can now be calculated considering the standard deviation of each concrete separately, in the equation-3. The number of samples to be tested for errors of 10%, 15% and 20% at the confidence level of 95% thus calculated using equation-3 and are given in Table 3.

It may be noted here that the number of samples so calculated do not follow any pattern with respect to the concrete strength. To check the validity of these estimated sample sizes, 4 samples of a new concrete designated as concrete-4 were tested. The average compressive strength of concrete-4 at the age of 28 days was 27.0 N/mm<sup>2</sup>. A comparison of the mean values of the parameters estimated from above test and their true values are summarized in Table 4. The estimated values of half length of the confidence interval 'd' calculated using equation-4, for n equal to 4 are also given side by side in Table 4.

TABLE 2  
Results of Statistical Analysis

Mix Designation	parameters	Mean ( $\mu$ )	SD ( $\sigma$ )	$C_v$ (%)	Distribution at $\alpha=5\%$
Concrete 1	p(%)	15.44	2.07	13.4	Normal
	$\ln(r_m)$	5.33	0.16	2.9	"
	R(%)	38.42	4.91	12.8	"
Concrete 2	p(%)	11.32	2.02	17.8	Normal
	$\ln(r_m)$	5.34	0.14	2.6	"
	R(%)	36.37	3.26	9.0	"
Concrete 3	P(%)	10.17	1.34	13.2	Normal
	$\ln(r_m)$	5.11	0.18	3.6	"
	R(%)	36.93	3.99	10.8	"

$\alpha$  = level of significance

The values of 'd' therefore are the expected deviations of the means from their true values. The true values of the parameters are estimated from the result given in Table 3 as described here after. An observation of the results given in Table 2 reveals that the porosity decreases consistently with increase in strength of concrete, but so far as  $\ln(r_m)$  and R are concerned, their variation with strength do not follow any particular pattern. Therefore, the true value of porosity for the concrete with 28 days cube strength of 27 N/mm<sup>2</sup> has been estimated by linear inter-

TABLE 3  
Number of Samples Size for Different % Errors

Designation	Parameters	n		
		E = 10%	E = 15%	E = 20%
Concrete 1	P(%)	7	4	2
	$\ln(r_m)$	1	1	1
	R(%)	7	3	2
Concrete 2	p(%)	12	6	4
	$\ln(r_m)$	1	1	1
	R(%)	4	2	1
Concrete 3	p(%)	7	3	2
	$\ln(r_m)$	1	1	1
	R(%)	5	2	2



TABLE 4  
Experimental Results and True Values for Concrete-4

Parameters	Mean - $\bar{x}$	True value $\mu$	Standard deviation	Deviations of mean from true value	'd' half length of confidence interval
P(%)	12.93	10.66	1.47	2.27	2.34
$\ln(r_m)$	5.28	5.26	0.01	0.02	0.02
R(%)	38.20	37.24	1.47	0.96	2.34

polation of porosities between the porosities of concrete with 28 days strengths of 18.8 and 33.1 N/mm<sup>2</sup>. However, for the other two parameters these true values have been assumed to be the mean of the three results given in Table 2. A comparison of the actual deviations with those expected show that the deviations are within the expected limit, and thus the true values obtained are reasonably accurate. So for the minimum numbers of samples are concerned, it can be inferred that when permissible error is say 15% the number sample to be tested shall be at least 6, 1 and 3 respectively for total porosity, log mean distribution radius and retention factors respectively as evident from the largest values of 'n' given in Table 3.

A comparison of these estimated samples sizes, with the minimum number of individual readings to be taken for various non-destructive and semi-destructive tests for concrete as stipulated in various standards and suggested in literature (23), reveals that, for most of the tests, the

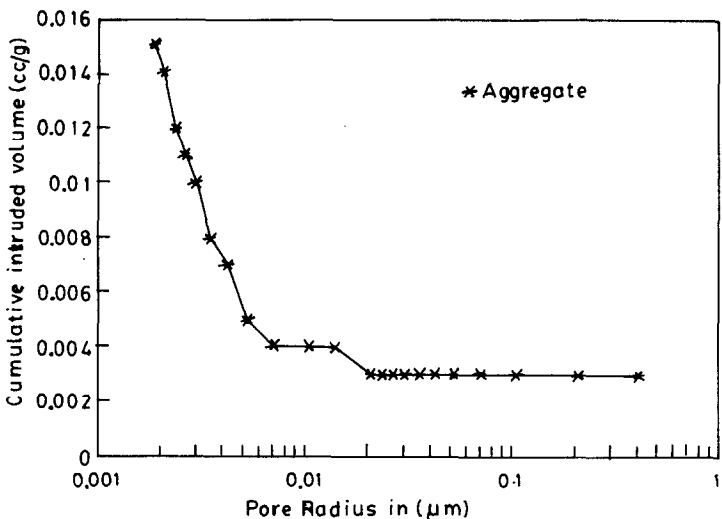


FIG. 4.  
PSD curve for aggregate only.

sample sizes are of the order of 3 or 6 for reasonable accuracy (15 to 20%) and are similar to what is being suggested in this paper for MIP.

### Form of the Sample for MIP

To investigate on the suitability of the form of sample that can be used in MIP study of concrete, porosimetry tests were conducted on two forms of samples taken from parent concrete, namely; mortar adhered with aggregate and mortar devoid of aggregate. These samples were obtained from core drilled from the floor slab of an actual building structure. Preparation of samples from the core followed the same procedure of crushing as described earlier for cubes. The number of samples however had to be restricted to 3 only and thus the expected accuracy of result is likely to be slightly lower than 20%. During the tests the first intrusion-extrusion cycle was immediately followed by a second cycle of intrusion-extrusion. On plotting together the three first intrusion curves so obtained for mortar of concrete devoid of aggregate, it was observed that the maximum difference between intrusions from, any two curves, at a given radius was of the order of 17.2%. The same maximum difference for mortar adhered with aggregate of concrete was 8.9%. Thus it was considered reasonable to represent all the intrusion curves in terms of two average intrusion curves only, one for mortar of concrete devoid of aggregate and another for the same mortar adhered with aggregate. This was achieved by averaging the intrusions of three samples at a particular radius and plotting the same against corresponding radius. In addition, pore size distribution of aggregate, extracted from the concrete, was also determined separately and is given in Fig. 4. Further, in order to normalize intrusion results

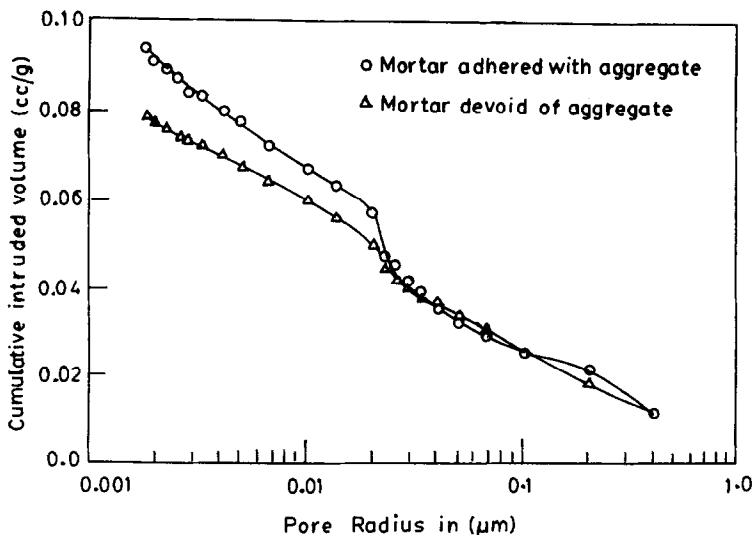


FIG. 5.

PSD curves for mortar adhered with and devoid of aggregate for 1st intrusion.

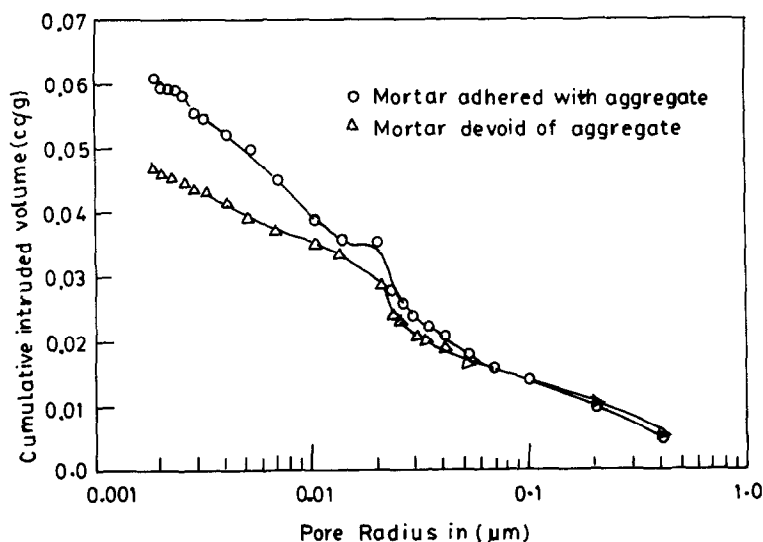


FIG. 6.

PSD curves for mortar adhered with and devoid of aggregate for 2nd intrusion.

of both forms of sample, the intrusion of aggregate was subtracted from the intrusion of mortar adhered with aggregate of concrete. The resultant intrusion was then divided by the weight of adhered mortar present in the sample to obtain the normalized intrusion per unit mass. The intrusion curves per unit mass for both forms of mortar sample obtained from the first cycle of intrusion curves are given in Fig. 5, whence it is observed that the total intrusion in mortar adhered with aggregate is more than that in mortar tested alone. Thus it appears that the mortar adhered with aggregate is more porous than mortar devoid of aggregate. This increase in porosity seems to belong to pore sizes greater than  $0.1\mu\text{m}$  and also in pore sizes smaller than  $0.03\mu\text{m}$ . Similar intrusion curves for the second cycle as given in Fig. 6. reveal that, mortar adhered with aggregate has higher intrusion than mortar devoid of aggregate in this case as well, but the pore sizes where intrusion was more, are less than  $0.04\mu\text{m}$ . The entrapped porosity represented by the difference in intrusion of first and second cycle for both forms of samples are shown in Fig. 7, and, they exhibit almost identical values of entrapped porosity at all radii except for a few points. The average retention for both mortar and concrete also appears to be same.

Values of porosity,  $\ln(r_m)$ , retention/gram and percentage retention for both forms of sample taken from the same in-situ concrete are given in Table 5 for comparison.

### Conclusions

1. The statistical distribution of some important parameters of MIP has been evaluated for various concrete mixes. The minimum number of tests to be conducted in order to estimate

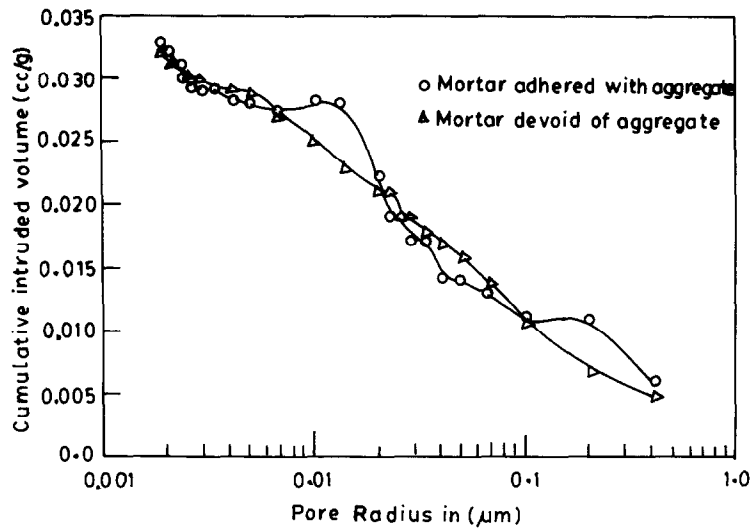


FIG. 7.  
PSD curves for entrapped porosity.

the values of these parameters experimentally, have also been specified for various percentages of errors and at the well accepted 95% confidence level.

- 2. Through study of porosity and pore size distribution of mortar adhered with aggregate and mortar devoid of aggregate, taken from the same parent concrete it has been demonstrated that mortar adhered with aggregate is more porous than mortar devoid of aggregate, although retention in cc per gram and  $\ln(r_m)$  log mean distribution radius may be similar. Thus it is suggested that mortar adhered with aggregate should be tested instead of mortar devoid of aggregate if properties of concrete is desired to be studied by MIP test.

TABLE 5  
Results of Mortar Adhered with Aggregate and Mortar Devoid of Aggregate

Parameters	Mortar adhered with aggregate	Mortar devoid of aggregate	% increase
p(%)	22.00	17.82	-19.0
$\ln(r_m)$	5.65	5.70	0.88
R(%)	30.00	41.20	37.33
Avg. Retention/gm	0.033	0.032	-3.03

### References

1. A. Auskern and W. Horn, *Journal of Testing and Evaluation*, JEVA, 1, 74 (1973).
2. I. Odler and M. Rößler, *CCR*, 15, 401 (1985).
3. D.H. Bager and E.J. Sellevold, *CCR*, 5, 171 (1975).
4. E.J. Sellevold, *CCR*, 5, 399 (1975).
5. R.F. Feldman and J.J. Beaudoin, *CCR*, 6, 389 (1976).
6. M. Rößler and I. Odler, *CCR*, 15, 320 (1985).
7. O.A. Kayyali, *Materials and Structures*, 20, 19 (1987).
8. R.S. Cook and R.S. Hover, *ACI Materials J.* March-April, 152 (1993).
9. N. Hearn and R.D. Hooton, *CCR*, 22, 970 (1992).
10. L. Konecny and S.J. Naqvi, *CCR*, 23, 1223 (1993).
11. D.N. Winslow and S. Diamond, *J. of Materials*, JMESA, 5, 56 (1970).
12. C. Atzeni, L. Massida and U. Sanna, *Proc. of 1st Int. RILEM Congress*, 195 (1987).
13. H.W. Reinhardt and K. Gaber, *Materials and Structures*, 23, 3(1990).
14. T. Allan, *Partical size Measurement*, Chapman and Hall, London (1981).
15. L. Tang, *CCR*, 16, 87 (1986).
16. F.H. Whittman, *Fracture Mechanics of Concrete*, p.56, Elsevier, The Netherlands (1983).
17. A. Carpenteri and A.R. Ingraffea, *Fracture Mechanics of Concrete, Materials Characterization and Testing*, Martinus Nijhoff Publishers, The Hague (1984).
18. J.P. Olliner and M. Maesat, *CCR*, 22, 503 (1992).
19. A.I. Lasker, M.Tech. Thesis, IIT Delhi, India (1993).
20. P.K. Mehta *Concretes: Structure, Properties and Materials*, Prentice-Hall Inc. N.J. (1985).
21. Y.W. Mai and B. Cotterell, *CCR*, 15, 945 (1985).
22. J. Miller and J.E. Freund, *Probability and Statistics for Engineers*, Prentice-Hall of India pvt. Ltd., Delhi (1981).
23. A.M. Leshchinsky, *Proc. of 2nd Int. RILEM Symp.*, 377 (1991).
24. R.G.D. Steel and J.H. Torrie, *Principles and Procedures of Statistics*, 2nd Edition, McGraw-Hill International Book Company, Singapore (1980).